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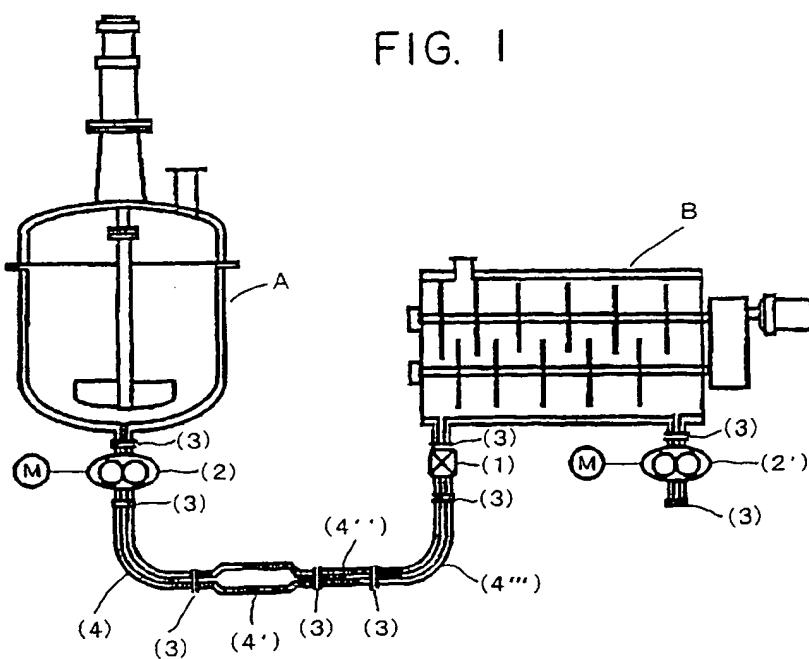
(54) Process for producing a polycarbonate resin

(57) A process for producing a polycarbonate resin through the melt polycondensation of an aromatic diol compound and a carbonic acid diester compound, wherein

(1) valves, (2) gear pumps, (3) flanges and (4) flow passages having the specified structures and characteristic properties are used in the production process.

According to the present invention, a high-quality polycarbonate can be obtained by an industrial process.

FIG. 1



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DescriptionDetailed Description of the InventionField of the Invention

[0001] The present invention relates to a process for producing a polycarbonate resin and, specifically, to a process for producing a polycarbonate resin through a melt polycondensation reaction between an aromatic diol compound and a carbonic acid diester. More specifically, the present invention relates to improvements on a valve, gear pump, flange or flow passage for the transfer of a reaction mixture among a plurality of polymerization reactors in an industrial process for producing a polycarbonate resin through a melt polycondensation reaction. The improvements in the present invention are applied to the discharge of a polycarbonate resin from a final polymerization reactor and the transfer of the polycarbonate resin to the following step.

5 increased in accordance with resistance (pressure loss) generated in the pipe (flow passage) to transfer the reaction mixture through the pipe, and these transfer members must stand high pressure.

10 **[0005]** The term "reaction mixture" as used herein means a mixture obtained after a polycondensation reaction starts, proceeds or ends in the step of obtaining an aromatic polycarbonate by carrying out the melt polycondensation reaction of a mixture containing an aromatic diol compound and an aromatic carbonic acid diester as essential ingredients in the presence of an ester exchange catalyst. A reaction mixture whose polymerization degree proceeds to some extent is generally called "prepolymer" according to chemical terminology

15 and a reaction mixture whose polymerization degree proceeds further is called "polymer" according to chemical terminology.

Brief Description of the Drawings**[0006]**

20 Fig. 1 is a diagram of a process typically showing an example of the arrangement of valves, pumps, flanges and flow passages for the production of the polycarbonate resin of the present invention; Fig. 2 is a diagram typically showing the transfer route of a reaction mixture in which valves, pumps, flanges and flow passages can be arranged in polymerization reactors (I to IV) for the production of the polycarbonate resin of the present invention (portions shown by dotted lines indicate the transfer route):

25 Explanations of notations in Fig. 1 and Fig. 2

- 30 (1) valve
- (2) gear pump
- (3) flange
- 35 (4) flow passage

40 I to IV: polymerization reactor

A: vertical polymerization reactor

B: horizontal polymerization reactor

45 Fig. 3 is a sectional view of an example of a ball valve of the prior art;

50 Fig. 4 is a sectional view of an example of a plug valve of the prior art;

Fig. 5 is a sectional view of an example of a T-shaped globe valve of the prior art;

55 Fig. 6 is a sectional view of an example of a Y-shaped globe valve according to the present invention;

Fig. 7 is a sectional view of an example of a Y-shaped globe valve according to the present invention;

Fig. 8 is a sectional view of an example of a valve according to the present invention;

Prior Art

[0002] An aromatic polycarbonate resin is widely used as a molding material because it is excellent in mechanical properties such as impact resistance as well as heat resistance and transparency. Known processes for producing such an aromatic polycarbonate resin include one (interfacial process) in which an aromatic diol compound such as bisphenol and phosgene are directly reacted with each other and one (melting process) in which an aromatic diol compound such as bisphenol and an aromatic carbonic acid diester such as diphenyl carbonate are subjected to an ester exchange reaction.

[0003] Out of these production processes, the process for producing a polycarbonate through an ester exchange reaction between an aromatic diol compound and an aromatic carbonic acid diester is generally carried out by distilling off from a reaction mixture a mono-hydroxy compound such as a phenol by-produced by gradually elevating the reaction temperature from 150°C which is the lowest temperature required to start an ester exchange reaction to about 350°C in the presence of an organic acid salt, inorganic acid salt, oxide, hydroxide, hydride or alcoholate of a metal as a catalyst and gradually reducing the pressure from atmospheric pressure to 13.3×10^{-6} MPa.

[0004] In the step of transferring the reaction mixture between polymerization reactors and the step of discharging the reaction mixture from the final polymerization reactor, valves, pumps, flanges and flow passages (pipes) are used. These transfer members are used at a high temperature and a high degree of vacuum. Since the viscosity of the reaction mixture rises to an extremely high level, generally 200 to 10,000 Pa·S (2,000 to 100,000 poise), which differs according to reaction conditions, along with the proceeding of the reaction (reaching completion), the pressure of the pump must be in-

Fig. 9 is a sectional view of an example of a valve according to the present invention
 Explanations of notations in Fig. 3 to Fig. 9

1	ball	5
2	inner space of ball	
3	portion opposite to ball 1	
4	clearance	
5	sheet packing	
11	plug	10
12	inner space	
13	valve body	
14	space	
31	stem	
32	disk	15
33	residence space	
34	seat	
41	stem	
42	disk	20
43	seat	
44	handle	
45	space	
51	valve body	
52	heating medium jacket	
53	tubular portion	25
54	inlet port of reaction mixture	
55	outlet port of reaction mixture	
56	stem	
57	gland packing	
58	handle	30
59	portion on a downstream side of spacer 62	
60	seat	
61	O ring	
62	spacer	
63	space	35
a	flow direction of fluid	
b	moving direction of plug	
c	flow direction of fluid	
d	flow direction of fluid	
e	flow direction of fluid	40

Fig. 10 is a transverse sectional view of an example of a gear pump according to the present invention;
 Fig. 11 is a transverse sectional view of an example of gear pump according to the present invention when seen from an axial direction;
 Fig. 12 shows a bushing fixed to a cover plate;
 Fig. 13 shows an example of the lubricating flow passage of a reaction mixture formed by a gear pump according to the present invention;
 Explanations of notations in Fig. 10 to Fig. 13

101	front plate	55
102	gear case	
103	back plate	
104	bushing	
105	heating medium jacket	
106	driving shaft	

107	follow shaft
108	gear
109	gear
110	inlet port of reaction mixture
111	outlet port of reaction mixture
112	driving shaft seal
113	O ring
114	scraper
115	bushing fixing bolt
116	O ring
117	lubricating groove
118	exhaust groove
119	sliding surfaces of gears 108 and 109
120	contact surfaces between bushing 104 and front plate 101 and between bushing 104 and back plate 103
121	inlet port of reaction mixture for lubrication

Fig. 14 is a sectional view of an example of a flange according to the present invention;
 Fig. 15 is a sectional view of an example of a flange according to the present invention;
 Explanations of notations in Fig. 14 to Fig. 15

201	flange (female)
202	flange (male)
203	heating medium jacket
204	pipe welded portion
205	metal-to-metal sealing surface
206	metal O ring attachment portion
207	groove portion (male)
208	groove portion (female)
209	bolt/nut attachment hole

Fig. 16 is a sectional view of an example of a socket according to the present invention;
 Explanations of notations in Fig. 16

301	pipe
302	pipe
303	socket
304	welding
305	liquid contact portion
306	surfaces in contact with socket 303 of pipes 301 and 302
307	surfaces in contact with pipes 301 and 302 of socket 303

Fig. 17 shows an example of a reducer (concentric type) in a flow passage according to the present invention;

Fig. 18 shows an example of a reducer (concentric type) in a flow passage according to the present invention;

Fig. 19 shows an example of a reducer (eccentric type) in a flow passage according to the present invention;

Fig. 20 shows an example of a reducer (eccentric

type) in a flow passage according to the present invention; and

Fig. 21 shows an example of a curved pipe portion in a flow passage according to the present invention.

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Explanations of notations in Fig. 17 to Fig. 21

401	reducer body	
402	small pipe side	
403	large pipe side	10
404	liquid contact portion	
405	welded portion	
406	curved portion	

[0007] It is an object of the present invention to improve (1) valves, (2) pumps, (3) flanges and (4) flow passages (may be referred to as "transfer members" hereinafter) used to transfer a reaction mixture when a polycarbonate resin (to be simply referred to as "polycarbonate" hereinafter) is produced by an industrial process. Particularly, it is an object of the present invention to make improvements to obtain a high-quality polycarbonate by suppressing as much as possible a deteriorated product formed by the residence of a reaction mixture in the transfer members.

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[0008] According to studies conducted by the inventors of the present invention, it has been found that the above objects of the present invention are attained by using (1) valves, (2) pumps, (3) flanges and (4) flow passages having the following structures and characteristic properties.

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[0009] That is, according to the present invention, there is provided a process for producing a polycarbonate resin through the melt polycondensation of an aromatic diol compound and a carbonic acid diester compound, wherein (1) valves, (2) pumps, (3) flanges and (4) flow passages having the following structures and characteristic properties are used in the production process.

25

(1) valve; Y-shaped globe valve which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less and a pressure durability of 0.1 MPa or more, and whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound

30

[0010] The polycarbonate production process of the present invention will be described in detail hereinafter.

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[0011] In the present invention, the transfer members are used to transfer a reaction mixture between polymerization reactors or to discharge the reaction mixture from a final polymerization reactor in the production of

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a polycarbonate through melt polycondensation. The transfer members will be described with reference to Fig. 1 and Fig. 2. Fig. 1 shows an example of the process of the present invention in which a vertical polymerization reactor A and a horizontal polymerization reactor B

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are used. In Fig. 1, a reaction mixture is discharged quantitatively by a gear pump (2) from the vertical polymerization reactor A, introduced into curved pipes (4') and (4'') through a curved pipe, passed through a curved pipe (4) and a valve (1) and introduced into the inlet of the horizontal polymerization reactor B. The reaction mixture is discharged from the outlet of the horizontal polymerization reactor B by a gear pump (2'). Flanges (3) are used in this transfer route.

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[0012] Fig. 1 is a diagram typically showing an example of the arrangement of the valves (1), gear pumps (2), flanges (3) and flow passages (4) of the present invention. The types of polymerization reactors, the arrangement order of the transfer members and the number of transfer members of each type are not limited to those of Fig. 1.

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[0013] The transfer members including pipes in Fig. 1 are heated by a heating medium jacket.

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[0014] Fig. 2 shows a flow of the reaction mixture

has such a structure that the reaction mixture under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less can be discharged and that the reaction mixture for lubricating contact portions among a gear, shaft and other gear pump members does not return into the pump and is discharged to the outside of the pump. (3) flange: flange which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less and a pressure durability of 0.1 MPa or more, whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound, and which has a heating medium jacket therein.

(4) flow passage; flow passage which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less and a pressure durability of 0.1 MPa or more, whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound, which is restricted and/or expanded at a vertical angle of 20° or less, and whose surface is smooth without level differences.

[0010] The polycarbonate production process of the present invention will be described in detail hereinafter.

[0011] In the present invention, the transfer members are used to transfer a reaction mixture between polymerization reactors or to discharge the reaction mixture from a final polymerization reactor in the production of

a polycarbonate through melt polycondensation. The transfer members will be described with reference to Fig. 1 and Fig. 2. Fig. 1 shows an example of the process of the present invention in which a vertical polymerization reactor A and a horizontal polymerization reactor B

are used. In Fig. 1, a reaction mixture is discharged quantitatively by a gear pump (2) from the vertical polymerization reactor A, introduced into curved pipes (4') and (4'') through a curved pipe, passed through a curved pipe (4) and a valve (1) and introduced into the inlet of the horizontal polymerization reactor B. The reaction mixture is discharged from the outlet of the horizontal polymerization reactor B by a gear pump (2'). Flanges (3) are used in this transfer route.

[0012] Fig. 1 is a diagram typically showing an example of the arrangement of the valves (1), gear pumps (2), flanges (3) and flow passages (4) of the present invention. The types of polymerization reactors, the arrangement order of the transfer members and the number of transfer members of each type are not limited to those of Fig. 1.

[0013] The transfer members including pipes in Fig. 1 are heated by a heating medium jacket.

[0014] Fig. 2 shows a flow of the reaction mixture

when polymerization reactors I to IV are connected in series. The transfer members of the present invention can be arranged along the route shown by dotted lines in Fig. 2. The polymerization reactors I to IV may be of either a vertical or horizontal type.

[0015] Fig. 2 shows four polymerization reactors but the number of polymerization reactors may be 2 to 5 and each of the polymerization reactors is maintained at a predetermined pressure and a predetermined temperature.

[0016] The present invention makes it possible to obtain a high-quality polycarbonate which rarely produces a deteriorated product of a polymer and is used as a base material for an optical recording material by using (1) valves, (2) gear pumps, (3) flanges and (4) flow passages having the above structures and characteristic properties as transfer members.

[0017] According to another aspect of the present invention, there is provided (a) a process for producing a polycarbonate resin through the melt polycondensation of an aromatic diol compound and a carbonic acid diester compound, wherein (1) valves, (2) gear pumps and (3) flanges used in the production process have the above structures and characteristic properties.

[0018] According to still another aspect of the present invention, there is provided (b) a process for producing a polycarbonate resin through the melt polycondensation of an aromatic diol compound and a carbonic acid diester compound, wherein at least two members out of (1) valves, (2) gear pumps and (3) flanges used in the production process have the above structures and characteristic properties.

[0019] In this aspect (b), (4) flow passages having the above structure and characteristic properties may be used.

[0020] According to a further aspect of the present invention, there is provided (c) a process for producing a polycarbonate resin through the melt polycondensation of an aromatic diol compound and a carbonic acid diester compound, wherein at least one member out of (1) valves, (2) gear pumps and (3) flanges used in the production process has the above structure and characteristic properties.

[0021] In the above aspect (c), (4) flow passages having the above structure and characteristic properties may further be used.

[0022] According to a still further aspect of the present invention, there is provided (d) a process for producing a polycarbonate resin through the melt polycondensation of an aromatic diol compound and a carbonic acid diester compound, wherein at least one member out of (1) valves, (2) gear pumps, (3) flanges and (4) flow passages used in the production process has the above structure and characteristic properties.

[0023] In this aspect (d), it is preferred that at least two out of the above transfer members used in the production process have the above structures and characteristic properties.

[0024] The polycarbonate in the present invention will be described in detail hereinafter.

[0025] The term "aromatic polycarbonate" as used herein means an aromatic polycarbonate obtained by 5 the melt polycondensation of an aromatic diol compound and a carbonic acid diester as essential ingredients in the presence of an ester exchange catalyst such as one comprising an alkali earth metal compound, an alkali metal compound or a nitrogen-containing basic 10 compound.

[0026] Illustrative examples of the aromatic diol compound include bis(4-hydroxyphenyl)methane, 2,2-bis (4-hydroxyphenyl)propane, 2,2-bis(4-hydroxy-3-methylphenyl)propane, 4,4-bis(4-hydroxyphenyl)heptane, 15 2,2-bis(4-hydroxy-3,5-dichlorophenyl)propane, 2,2-bis (4-hydroxy-3,5-dibromophenyl)propane, 1,1-bis(4-hydroxyphenyl)-3,3,5-trimethylcyclohexane, 4,4-(m-phenylenediisopropylidene)diphenol, bis(4-hydroxyphenyl) oxide, bis(3,5-dichloro-4-hydroxyphenyl)oxide, p,p'-di 20 hydroxydiphenyl, 3,3-dichloro-4,4'-dihydroxydiphenyl, bis(hydroxyphenyl)sulfone, resorcinol, hydroquinone, 1,4-dihydroxy-2,5-dichlorobenzene, 1,4-dihydroxy-3-methylbenzene, bis(4-hydroxyphenyl)sulfide, bis (4-hydroxyphenyl)sulfoxide and the like. Out of these, 25 2,2-bis(4-hydroxyphenyl)propane is particularly preferred.

[0027] Illustrative examples of the carbonic acid diester include diphenyl carbonate, ditolyl carbonate, bis (chlorophenyl)carbonate, m-cresyl carbonate, dinaphthyl carbonate, bis(diphenyl)carbonate, dimethyl carbonate, diethyl carbonate, dibutyl carbonate, dicyclohexyl carbonate and the like. Out of these, diphenyl carbonate is particularly preferred.

[0028] Further, the polycarbonate of the present invention may further contain an aliphatic diol such as ethylene glycol, 1,4-butanediol, 1,4-cyclohexane dimethanol or 1,10-decanediol; a dicarboxylic acid such as succinic acid, isophthalic acid, 2,6-naphthalenedicarboxylic acid, adipic acid, cyclohexanecarboxylic acid or terephthalic acid; or an oxyacid such as lactic acid, p-hydroxybenzoic acid or 6-hydroxy-2-naphthoic acid as required.

[0029] The alkali metal compound used as a catalyst is, for example, a hydroxide, bicarbonate, carbonate, acetate, nitrate, nitrite, sulfite, cyanate, thiocyanate, 45 stearate, borohydride, benzoate, hydrogen phosphate, bisphenol salt or phenol salt of an alkali metal.

[0030] Illustrative examples of the alkali metal compound include sodium hydroxide, potassium hydroxide, lithium hydroxide, sodium bicarbonate, potassium bicarbonate, lithium bicarbonate, sodium carbonate, potassium carbonate, lithium carbonate, sodium acetate, potassium acetate, lithium acetate, sodium nitrate, potassium nitrate, lithium nitrate, sodium nitrite, potassium nitrite, lithium nitrite, sodium sulfite, potassium sulfite, lithium sulfite, sodium cyanate, potassium cyanate, lithium cyanate, sodium thiocyanate, potassium thiocyanate, lithium thiocyanate, sodium stearate, potassium stearate, lithium stearate, sodium borohydride, potassium

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borohydride, lithium borohydride, sodium phenyl borate, potassium phenyl borate, lithium phenyl borate, sodium benzoate, potassium benzoate, lithium benzoate, disodium hydrogen phosphate, dipotassium hydrogen phosphate, dilithium hydrogen phosphate, disodium salts, dipotassium salts and dilithium salts of bisphenol A, and sodium salts, potassium salts and lithium salts of phenol.

[0031] The alkali earth metal compound used as a catalyst is a hydroxide, acetate, nitrate, nitrite, sulfite, cyanate, thiocyanate, stearate, benzoate, hydrogenphosphate, bisphenol salt or phenol salt of an alkali earth metal.

[0032] Illustrative examples of the alkali earth metal compound include barium hydroxide, calcium hydroxide, strontium hydroxide, magnesium hydroxide, barium acetate, calcium acetate, strontium acetate, magnesium acetate, barium nitrate, calcium nitrate, strontium nitrate, magnesium nitrate, calcium salts, barium salts, strontium salts and magnesium salts of bisphenol A, calcium salts, barium salts, strontium salts and magnesium salts of phenol, and the like.

[0033] The alkali metal compound or alkali earth metal compound as a catalyst is preferably used in such a proportion that the amount of an elemental alkali metal or alkali earth metal contained in the catalyst should be 1×10^{-8} to 5×10^{-5} equivalent, preferably 5×10^{-7} to 1×10^{-5} equivalent based on 1 mol of the aromatic diol compound.

[0034] Illustrative examples of the nitrogen-containing basic compound as a catalyst include ammonium hydroxides having an alkyl, aryl or alkylaryl group such as tetramethyl ammonium hydroxide (Me_4NOH), tetraethylammonium hydroxide (Et_4NOH), tetrabutylammonium hydroxide (Bu_4NOH), benzyltrimethyl ammonium hydroxide ($\phi\text{-CH}_2(\text{Me})_3\text{NOH}$) and hexadecyltrimethyl ammonium hydroxide; tertiary amines such as triethylamine, tributylamine, dimethylbenzylamine and hexadecyltrimethylamine; and basic salts such as tetramethyl ammonium borohydride (Me_4NBH_4), tetrabutyl ammonium borohydride (Bu_4NBH_4), tetrabutyl ammonium tetraphenyl borate (Me_4NBPh_4) and tetrabutyl ammonium tetraphenyl borate (Bu_4NBPh_4).

[0035] The above nitrogen-containing basic compound is preferably used in such a proportion that the amount of ammonium nitrogen atoms contained in the nitrogen-containing basic compound should be 1×10^{-5} to 5×10^{-4} equivalent, more preferably 2×10^{-5} to 5×10^{-4} equivalent, particularly preferably 5×10^{-5} to 5×10^{-4} equivalent based on 1 mol of the aromatic diol compound.

[0036] In the present invention, (a) an alkali metal salt of a complex of the group 14 element of the periodic table or (b) an alkali metal salt of an oxoacid of the group 14 element of the periodic table as disclosed by JP-A 7-268091 (the term "JP-A" as used herein means an "unexamined published Japanese patent application") may be used as the alkali metal compound catalyst as

desired. The group 14 element of the periodic table is silicon, germanium or tin.

[0037] The polycondensation reaction can be quickly and fully promoted by using the alkali metal salt of the above mentioned complex of the group 14 element as a polycondensation reaction catalyst. Further, an undesired side-reaction such as a branching reaction generated during the polycondensation reaction can be suppressed to a low level by using the above catalyst.

[0038] The above polycondensation reaction catalyst is preferably used in such a proportion that the amount of an elemental alkali metal contained in the catalyst should be 1×10^{-8} to 5×10^{-5} equivalent, more preferably 5×10^{-7} to 1×10^{-5} equivalent based on 1 mol of the aromatic diol compound.

[0039] At least one co-catalyst selected from the group consisting of oxoacids of the group 14 elements of the periodic table and oxides of the elements may be used in conjunction with the above catalyst as required in the polycondensation reaction of the present invention.

[0040] A branching reaction which is readily initiated during the polycondensation reaction, the formation of foreign matter in an apparatus during molding and an undesired side-reaction such as a color difference can be effectively suppressed without impeding the capping reaction of a terminal and decreasing a polycondensation reaction speed by using the above co-catalyst in a specific proportion.

[0041] The co-catalyst is preferably existent in such a proportion that the amount of the group 14 metal element of the periodic table contained in the co-catalyst should be 0.1 to 30 mols based on 1 mol (atoms) of the elemental alkali metal contained in the polycondensation reaction catalyst.

[0042] The above catalytic system has such an advantage that a polycondensation reaction and a terminal capping reaction can be quickly and fully promoted by using these catalysts for the polycondensation reaction.

[0043] It can also suppress an undesired side-reaction such as a branching reaction in the polycondensation reaction system to a low level.

[0044] The temperature and the pressure for carrying out an ester exchange reaction between an aromatic diol compound and a carbonic acid diester compound are not particularly limited in the present invention. Any temperature and any pressure are acceptable if the reaction starts and a monohydroxy compound formed by the reaction is swiftly removed from the reaction system. However, it is the most common that, after the reaction is started at a temperature of 150 to 200°C and a pressure of $40,000 \times 10^{-6}$ to $13,333 \times 10^{-6}$ MPa, the reaction temperature is elevated and the reaction pressure is reduced as the molecular weight of a polycarbonate grows

[0045] along with the proceeding of the reaction and the reaction is carried out finally at a temperature of 270 to 350°C and a pressure of 133×10^{-6} MPa or less. More specifically, it is preferred to carry out the reaction at a tem-

perature of 150 to 220°C and a pressure of 40,000 x 10⁻⁶ to 13,333 x 10⁻⁶ MPa when the viscosity average molecular weight (M_v) of a polycarbonate is 1,000 to 2,000, at a temperature of 180 to 240°C and a pressure of 20,000 x 10⁻⁶ to 2,000 x 10⁻⁶ MPa when M_v is 2,000 to 4,000, at a temperature of 200 to 250°C and a pressure of 13,333 x 10⁻⁶ to 1,333 x 10⁻⁶ MPa when M_v is 4,000 to 6,000, at a temperature of 220 to 280°C and a pressure of 4,000 x 10⁻⁶ to 133 x 10⁻⁶ MPa when M_v is 6,000 to 10,000, and at a temperature of 250 to 300°C and a pressure of 133 x 10⁻⁶ MPa or less when M_v is more than 10,000. The unit of pressure used is absolute pressure throughout the specification unless otherwise stated.

[0044] A stabilizer may be added to the polycarbonate obtained in the present invention. Known stabilizers are effectively used as the stabilizer used in the present invention. Out of these, ammonium salts and phosphonium salts of sulfonic acid are preferred, and the above salts of dodecylbenzenesulfonic acid such as tetrabutylphosphonium dodecylbenzenesulfonate and the above salts of paratoluenesulfonic acids such as tetrabutylammonium paratoluenesulfonate are particularly preferred. Preferred sulfonic acid esters include methyl benzene sulfonate, ethyl benzene sulfonate, butyl benzene sulfonate, octyl benzene sulfonate, phenyl benzene sulfonate, methyl paratoluene sulfonate, ethyl paratoluene sulfonate, butyl paratoluene sulfonate, octyl paratoluene sulfonate, phenyl paratoluene sulfonate and the like. Out of these, tetrabutyl phosphonium dodecylbenzenesulfonate is the most preferred.

[0045] The amount of the stabilizer is 0.5 to 50 mols, preferably 0.5 to 10 mols, more preferably 0.8 to 5 mols based on 1 mol of the polymerization catalyst selected from alkali metal compounds and/or alkali earth metal compounds.

[0046] Equipment for carrying out the present invention is not particularly limited and the present invention may be carried out in a batch or continuous system. When the batch system is used, two reactors are generally connected in series, an agitator equipped with a distillator is used for the first reactor, and an agitator without distillator is used for the second reactor to carry out a reaction under different conditions. In this case, it is preferred that both reactors be connected by a pipe equipped with a valve, the reaction mixture of the first reactor be transferred to the second reactor without being exposed to the outside air, and a reaction be carried out to a desired degree of polymerization in the second reactor.

[0047] When the continuous system is used, two or more reactors are generally connected in series, adjacent reactors are connected by a pipe equipped with a valve, equipment comprising a pump for transferring a reaction mixture as required is used to supply raw materials and a catalyst to the first reactor continuously while the reactors are maintained under different conditions, and a polycarbonate having a desired degree of

polymerization is discharged continuously from the final reactor.

[0048] The above equipment must have a function to stop or branch a flow of the reaction mixture and valves installed in vessels and pipes for this purpose play an important role.

[0049] Gear pumps, flanges and flow passages for transferring or discharging the reaction mixture must have an important function to obtain a high-quality polycarbonate.

[0050] According to studies conducted by the present inventors, it has been found that the polycarbonate is colored, is changed or is varried a deteriorated product called "three-dimensionally crosslinked gel" when it receives long-time heat history though the influence of oxygen is completely eliminated, unlike other polycondensation polymers such as polyethylene terephthalate. This reduces the quality of the obtained polycarbonate and causes a serious problem in optical application.

[0051] To solve this problem, it is important to eliminate a residence portion of the reaction mixture in the equipment. Various studies have been made to eliminate the residence portion in the reactor and various proposals have been made. However, the quality of a polycarbonate obtained by melt polymerization is still unsatisfactory in fact.

[0052] The expression "residence portion of the reaction mixture" as used herein means a portion of the reaction mixture which deteriorates the quality of a polycarbonate product because a deteriorated product formed at a site where the continuous replacement of the reaction mixture hardly occurs is contained in the reaction mixture. The expression "residence portion" means "residence portion of the reaction mixture" throughout the specification.

[0053] As described above, in the present invention, when a polycarbonate is produced by an industrial process, it is an object of the present invention to improve and change each of (1) valves, (2) pumps, (3) flanges and (4) flow passages (they may be referred to as "transfer members") for the transfer of a reaction mixture. The problems of each of the transfer members of the prior art and the improvements of the transfer members made by the present invention will be described in detail hereinafter.

(1) valve;

[0054] In the production of a polycarbonate through a melt polycondensation reaction, a ball valve, plug valve or T-shaped globe valve has been used to stop a flow of a reaction mixture in a straight portion of a pipe.

[0055] However, the significance of the influence of the valve upon the quality of a polycarbonate product has not been realized in the prior art and these valves have been selected from the viewpoints of a pressure loss generated in the pipe, pressure durability, vacuum resistance and the economy of equipment investment.

[0056] The present inventors have found in the course of studies on the improvement of the quality of a polycarbonate product produced by an ester exchange method that the structure of a valve has a great influence upon the quality of a polycarbonate product and have conducted intensive studies on the structures of various valves.

[0057] It seems that the reason why this problem has not been realized in the production of a polycarbonate by an interfacial method is that temperature, pressure and other conditions are much milder than those of the ester exchange method.

[0058] The problems of the valve of the prior art which have been found in the course of studies are as follows.

(1) example of ball valve (see Fig. 3)

[0059] In Fig. 3, a ball valve has such a structure that a passing fluid can flow when the inner space 2 of a ball 1 is opened in a flow direction "a" of the fluid as shown in Fig. 3 and its flow is stopped when the inner space 2 is not opened in the flow direction "a" of the fluid.

[0060] It has been found that, in the valve having the above structure, when a flow of a reaction mixture is stopped, the reaction mixture existent in the inner space 2 of the ball 1 is cooped up in the inner space 2 and the cooped and retained reaction mixture is apt to deteriorate and pollute the quality of a polycarbonate product intermittently by opening the ball valve. It has also been found that as a clearance 4 for moving the valve is interposed between the ball 1 and a portion 3 opposite to the ball 1 of the valve body, the reaction mixture enters this clearance, resides in the clearance and deteriorates, making the movement of the valve difficult and generating highly deteriorated foreign matter losing flowability (gel) with the result that a non-negligible quality problem arises in the polycarbonate product. The clearance 4 above and below the ball is shown in Fig. 3 but it also exists around side portions of the ball 1. Since the ball 1 slides over a sheet packing 5 while it contacts it during the operation of the valve, the surface of the seat packing 5 is apt to be chipped and generate powders. Particularly when a heat durability carbon packing is used as the seat packing 5, generated powders have a significant bad influence upon the quality of a product.

(2) example of plug valve (see Fig. 4)

[0061] This valve has such a structure as shown in Fig. 4 and is superior to a ball valve because it does not generate packing powders as it is not sealed with a seat packing unlike the above ball valve, its maintenance is easy as it does not use a seat packing having a shorter service life than the valve body and there is no residence portion as there is no clearance between a plug 11 and a valve body 13. However, like the ball valve, as shown in Fig. 4, when the flow of the reaction mixture is stopped, the reaction mixture is cooped in the inner

space 12 of the plug 11 and the cooped and retained reaction mixture is liable to deteriorate. Further, to move the valve, the plug 11 must be turned after the plug is lifted to form a clearance (the plug itself is moved in a direction of the handle of the valve, that is, direction "b" in Fig. 4) because metal-to-metal sealing is effected for a gap between the valve body 13 and the plug 11. By this operation, a space 14 which the reaction mixture enters is formed around the side surface of the plug and the reaction mixture residing in this portion is apt to deteriorate. Further, it is difficult to maintain a vacuum atmosphere in the valve when the valve is moved, the reaction mixture is liable to contact air outside the valve and deteriorate by oxidation, and the automation of the operation of lifting this plug is difficult.

[0062] The present inventors have conducted studies on the structure and characteristic properties of a valve for improving the above problems of the prior art and efficiently obtaining a polymer which rarely produces a deteriorated product and has excellent quality in an industrial process for producing a polycarbonate. As a result, they have found that a high-quality polycarbonate which rarely produces the deteriorated product of the polymer can be obtained by using the following valve.

[0063] That is, according to the present invention, there is provided a process for producing a polycarbonate resin through the melt polycondensation of an aromatic diol compound and a carbonic acid diester compound, wherein (1) valves having the following structure and performance are used in the production process.

(1) valve: Y-shaped globe valve which has substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less, and whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound.

[0064] A detailed description is subsequently given of the above valve.

[0065] In the valve of the present invention, the expression "substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less" means that the leakage of gas from the outside of the valve is 120×10^{-6} MPa·l/h or less based on 1 liter of the inner capacity of the valve when the ultimate vacuum degree of the valve is 13.3×10^{-6} MPa.

[0066] With this method for measuring the leakage of gas, the measurement may not be easy because the inner capacity of the valve is too small. In this case, when a helium leak test is carried out at 0.133×10^{-6} MPa, 3×10^{-5} ACC/S or less can be used instead. The measurement method is as follows.

55 a. gas leakage measurement method

[0067] The measurement of the leakage of gas is carried out by sucking gas inside the valve by a vacuum

pump to reduce the pressure inside the value to 13.3×10^{-6} MPa, stopping suction and confirming after one hour that the pressure inside the value is lower than a predetermined pressure. When the capacity of the value is 1 liter, for example, the value stands the test the pressure inside the value is lower than 133×10^{-6} MPa one hour after the suction of the vacuum pump is stopped.

b. helium leak test

[0068] Gas inside the value is sucked by a vacuum pump to reduce the pressure inside the value to 0.133×10^{-6} MPa or less, a helium leak detector is connected to the value and the vacuum pump, the value is covered with a bag while suction by the vacuum pump is continued, helium is blown into the inside of bag from the outside, and the leakage of helium into the inside of the value is measured by the helium leak detector. If the leakage of the detected helium is 3×10^{-5} ACC/S or less, the value stands test. The DLMS-TP3E of Nippon Shinku Gijutsu Co., Ltd. may be used as the helium leak detector.

[0069] ACC/S means atm·cc/sec which indicates a volume at 1 atm. and normal temperature as the leakage of helium per unit sec.

[0070] Thus, the valve is desired to have no leakage because it is the outside air, especially oxygen entering the reactor that deteriorates the reaction mixture and has a significant bad influence upon the quality of a polycarbonate product. Since the valve has a relatively complex structure that it has a movable portion, such air easily enters the valve.

[0071] Since the reaction mixture is oxidized as soon as it is exposed to oxygen, it is colored or deteriorated, thereby greatly reducing the quality of the polycarbonate. Therefore, it is important to prevent the reaction mixture from being exposed to oxygen contained in the air and it is desired to improve airtightness and place the gland portion of the valve in 98 vol% or more of nitrogen atmosphere.

[0072] In the valve of the present invention, the expression "liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound" means that any material is acceptable as the material of the liquid contact portion if it does not impair the function of the valve and does not substantially dissolve into the product during the use of the valve. A seat portion of the valve is preferably made from stellite such as ST-ELLITE FACE#6 and the valve body is preferably made from stainless steel such as SUS304, SUS304L, SUS316, SUS316L, SUS630, SCS13, SCS14, SCS16 or SCS19, specified by JIS.

[0073] Any material is acceptable as the material of the gland packing if it has required sealing properties. For example, a coil packing prepared by knitting asbestos, carbon fibers, polytetrafluoroethylene (PTFE) fib-

ers, aramide fibers or expanded graphite yarn into a pre-determined shape with a rectangular cross section or the like and impregnating with PTFE, graphite or lubricating oil and/or a packing molded into a ring form with a V-shaped cross section are/is preferably used.

[0074] Any material is acceptable as the material of a gasket if it has required sealing properties during the use of the valve. A spiral wound gasket prepared by placing a filler such as asbestos, inorganic paper, expanded graphite or PTFE upon a metal hoop having a V-shaped cross section and spirally winding the laminate under tension is preferably used.

[0075] In the present invention, the Y-shaped globe valve is such as shown in Fig. 6 that a pipe and the stem 15 of the valve are arranged into the form of letter Y. When the valve is opened, the stem can move not to disturb a flow of a reaction mixture in the pipe as much as possible.

[0076] The present inventors have paid attention to a globe valve as a valve which has a favorable influence upon the quality of a polycarbonate product.

[0077] However, in a T-shaped globe valve of the prior art whose stem is moved in a direction perpendicular to a flow passage at the reaction mixture inlet port and outlet port of the valve, the problem of the reaction mixture residing in the above ball valve and plug valve and a residence portion in the clearance is slightly improved. Since the T-shaped globe valve has such a structure as shown in Fig. 5, it has a disk 32 having a larger diameter 25 than that of a stem 31 at the end of the stem, a large residence space 33 is formed behind the disk 32, the reaction mixture in this residence space 33 becomes a residence portion which rarely receives the influence of a flow of a fluid (an upper portion of the residence space 33 of Fig. 5) unlike the ball valve and the plug valve, the cross section of the flow passage in the valve is greatly changed by the existence of a seat 34 which receives the disk 32, and the flow of the fluid greatly changes its direction as shown by an arrow "c". Therefore, it has 30 been found that the T-shaped globe valve has such a problem that the flow is disturbed, thereby producing a great pressure loss, and that this problem becomes more serious as the viscosity of the fluid increases.

[0078] In contrast to this, in the Y-shaped globe valve 45 shown in Fig. 6, a disk 42 is provided at the end of a stem 41 which is arranged at an angle to a flow direction of a reaction mixture, a flow of the passing fluid shown by an arrow "d" is stopped by contact between the disk 42 and a seat 43 provided in a fluid passing portion, the stem 41 and the disk 42 are moved in the direction of a handle 44 by turning the handle 4 in a certain direction, the flow of the passing fluid shown by the arrow "d" is resumed, the volume of a space 45 behind the disk 42 is reduced by the moving stem 41 and disk 42 at this 50 point, the disturbance of the flow of the reaction mixture is smaller than that of an ordinary T-shaped globe valve, and a pressure loss is small when the fluid passes through this portion, whereby the retained reaction mix-

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ture has small chances of going into the flow of the reaction mixture incidentally and the residence portion (portion corresponding to the space 33 in Fig. 5) shown by the space 45 formed behind the disk is small. Therefore, it has been found that this Y-shaped globe valve is suitable as a valve for stopping the polycarbonate.

[0079] When the valve has substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less and its liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound, it has been verified that the valve can stand long-time use of 1 year or more and produce good results in the maintenance of the quality of a polycarbonate product as a device for producing a polycarbonate.

[0080] However, even in this Y-shaped globe valve, it has been revealed that, when the valve is fully opened, a non-negligible residence portion remains behind the disk (space 45 in Fig. 6) and the effect of reducing a residence portion may be unsatisfactory for the quality of a product according to the use conditions of the valve such as temperature, flow rate and viscosity as a device for producing a polycarbonate. Especially in a larger valve, this residence portion causes greater bad influence upon the quality of a product.

[0081] The residence portion formed in these valves causes the deterioration of a polycarbonate such as coloration, crosslinking or gelation and may exert a non-negligible influence upon product quality. This is an especially serious problem to be solved for a polycarbonate which is liable to reside and deteriorate in the residence portion.

[0082] The present inventors have tackled with the further improvement of the Y-shaped globe valve through studies on these valves and have found that a Y-shaped globe valve having the following features can solve the above problems completely.

[0083] A preferred example of the valve of the present invention is shown in Fig. 7. The example shown in Fig. 7 is mere an embodiment of the present invention, and it should be understood that the present invention is not limited by this figure.

[0084] In Fig. 7, reference numeral 51 denotes a valve body. This valve body 51 is covered with a heating medium jacket 52 therearound. A reaction mixture inlet port 54 is provided at one end of the tubular portion 53 of the valve body 51 and a reaction mixture outlet port 55 is provided at the other end. A stem 56 is provided in a center portion of the valve body 51 and can be slid vertically by operating a handle 58 while it is sealed with a gland packing 57. A flow of a fluid is cut off when an end portion of the stem 56 contacts a seat 60. Closing the valve indicates this state and is shown by a solid line in Fig. 7. Meanwhile, fully opening the valve indicates the opposite state and is shown by a broken line in Fig. 7. The valve is fully opened to flow a reaction mixture.

[0085] The valve body 51 is made from the above cor-

rosion-resistant material and the stem 56 is also made from the same material. Since they are made from the same material, even if the temperatures of the valve body 51 and the stem 56 rise, the clearance between

5 them is maintained at a fixed value, rarely becomes larger than a designed value or suppresses contact between them because the coefficient of thermal expansion of the valve body 51 and the stem 56 are the same. This is preferred from the view point of reducing the residence portion. The seat 60 is made from the above sealing material such as STELLITE FACE#6 to prevent the seizure of the valve body 51 and the stem 56 and to enable sealing properties to be retained for a long time.

[0086] Reactors are connected by a pipe and the valve is generally installed in the pipe. In Fig. 7, the reaction mixture inlet port 54 and the reaction mixture outlet port 55 are butt welded (butt welded joint) to the pipe.

[0087] In Fig. 7, reference notation 54 denotes the reaction mixture inlet port and 55 the reaction mixture outlet port. It merely means that this arrangement is very common, and the reaction mixture inlet port and the reaction mixture outlet port may be installed in opposite directions according to the actual installation space.

[0088] The reaction mixture is supplied continuously 25 from the reaction mixture inlet port 54 by the valve thus constituted. When the valve is opened, the reaction mixture is discharged to the outside of the system from the reaction mixture outlet port 55. When the valve is closed, the reaction mixture is shut off and does not flow to the reaction mixture outlet port 55.

[0089] The above Y-shaped globe valve according to the present invention has such a great improvement that a residence portion behind the disk is eliminated to remove a space behind the disk because it has no disk at 35 the end of the stem and a portion into which the stem is inserted of the valve body and the stem have substantially the same thickness.

[0090] The expression "have substantially the same thickness" means that the thickness of the stem is smaller than that of the portion into which the stem is inserted of the valve body by a clearance required for the stem to slide in the valve body. The difference between the outer diameter of the stem and the inner diameter of the portion into which the stem is inserted of the valve body 40 is preferably set to 2 mm or less. The difference is more preferably 1 mm or less, much more preferably 0.5 mm or less.

[0091] The cross section of the portion into which the stem is inserted of the valve body is desirably $\pm 15\%$ of 45 the cross section of a portion through which the reaction mixture flows of the tubular portion to suppress the turbulent flow of the reaction mixture. It is more desirably the same as the inner diameter of the tubular portion.

[0092] In Fig. 8, a spacer 62 having a smooth curved surface so as not to disturb the flow line of the running reaction mixture is provided at the end of the stem 56.

[0093] An end of the spacer 62 is smoothly connected to a portion 59 on a downstream side of the spacer 62,

which is the inner surface of an upper portion of a tubular portion 53, when the valve is fully opened and the fluid flows in a direction "e" of Fig. 8. That is, the end of the spacer 62 which is the end portion of the stem of the Y-shaped globe valve and the inner surface of the upper portion of the tubular portion of the valve have a curved surface so that they can form a smooth curved surface when the valve is opened. The curved surface is shown by a broken line in Fig. 8. Other numerals in Fig. 8 signify the same elements as those of Fig. 5.

[0094] In the present invention, for example, the spacer 62 is provided at the end portion of the stem 56 to form a smooth curved surface not to disturb the flow of the reaction mixture. The formation of this smooth curved surface reduces the residence portion without disturbing the flow of the fluid as much as possible and can prevent the deteriorated polymer which caused in the residence portion which still exists in the present invention from flowing into the flow of the fluid by the turbulent flow of the fluid.

[0095] The Y-shaped globe valve shown in Fig. 7 does not have a structure that provides a smooth curved surface so as not to disturb the flow of the reaction mixture when it is fully opened. Therefore, the present invention shown in Fig. 8 has been accomplished based on the discovery of a new fact that this structure is one of the causes of deteriorating the quality of a polycarbonate product.

[0096] The expression "formation of this smooth curved surface" as used herein means the formation of a substantially smooth curved surface. For example, in Fig. 8, the diameter of the spacer 62 is made a little smaller than the diameter of the stem 56 to realize perfect contact between the stem 56 and the seat 60 when the valve is closed. Therefore, when the valve is closed, 1 to 0.5 mm space 63 is formed between the spacer 62 and the valve body 51. "The formation of a gentle curved surface" in the present invention comprehends a case where the smooth curved surface becomes partly discontinuous by the space 63.

[0097] Further, a clearance is required between the stem and the valve body for the stem to slide in the structure of the valve. It has been found that the provision of an O ring (denoted by notation 61 in Fig. 7 and Fig. 8) having excellent heat durability and corrosion resistance in this portion is more effective so that the stem can slide substantially in contact with the inner surface of the O ring. This can effectively prevent a retained product of the fluid from flowing into the reaction mixture, which can take place in the clearance as well. A preferred example of the O ring used for this purpose is an O ring made from a perfluoro elastomer (for example, Kalrez of Du Pont Dow Elastomers.)

[0098] Preferably, the Y-shaped globe valve of the present invention is uniformly heated by a heating medium jacket, electric heater or the like. In the case of a heating medium jacket, to integrate the heating medium jacket with the valve body as shown in Fig. 5 is more

preferred than to cover the Y-shaped globe valve of the present invention with the heating medium jacket from the viewpoint of uniform heating. When the Y-shaped globe valve of the present invention is relatively large in size, it is preferred to provide a baffle plate in the heating medium jacket or to increase the number of the flow passages of a heating medium so as to uniformly heat the Y-shaped globe valve of the present invention.

[0099] In the valve of the present invention, the surface roughness of the inner portion of the liquid contact portion is preferably 10 μm or less. This surface roughness is 10 μm or less, preferably 3.3 μm or less in terms of Rmax. For example, the liquid contact portion is finished with a buff of #200 or more, has a gloss higher than that of a standard sample and is free from pin holes, circular fine streaks, scratches, level differences and dimples. More preferably, the liquid contact portion has a surface roughness of 1.6 μm or less in terms of Rmax. That is, it is finished with a buff of #300 or more, or subjected to finishing such as honing, super finishing,lapping, liquid honing, chemical polishing or electrolytic polishing. In the present invention, Rmax was measured in accordance with JIS B0601-1982.

[0100] In the valve of the present invention, the term "heat durability" as used herein means that the airtightness and pressure durability of the valve are higher than required air tightness and pressure durability during operation even when the strength of the material at a high temperature becomes lower than a value at normal temperature and any problem does not arise in its use even when the valve receives force such as the thermal expansion of its material.

[0101] When a polycarbonate is produced, the heat durability temperature of the equipment is preferably set to a range of 150 to 350°C according to its operation conditions. When the heat durability temperature is higher than 350°C, the material is limited, its processing becomes difficult, the obtained valve becomes expensive, a wide clearance must be formed between the parts of the valve to eliminate the influence of the thermal expansion of the material constituting the valve, and the residence and deterioration of the reaction mixture in this portion readily occur disadvantageously.

[0102] The inside of the valve of the present invention preferably has a pressure durability of 0.1 MPa or more. The expression "pressure durability of 0.1 MPa or more" means that the valve does not leak a liquid or gas to the outside of the valve or to a secondary side and does not cause any problem at all in its use whether it is opened or closed, even when a predetermined pressure of 0.1 MPa or more is applied to the inside of the valve, when a side which is shut off by the stem and to which pressure is applied when the valve is closed is called "primary side" and a side to which pressure is not applied is called "secondary side". The pressure durability is generally in the range of 0.1 to 30 MPa. When the clearance near the sealing portion (surface for separating the primary side from the secondary side) of the stem of the

valve must be made wide in design (difference of diameter is 2 mm or more) to prevent leakage (also called "inner leakage") from the above primary side to the secondary side, the pressure durability of the primary side against the secondary side may be made lower than pressure durability against the outside of the valve.

[0103] Means of connecting the Y-shaped globe valve of the present invention to the pipe or the like preferably has the same pressure durability, corrosion resistance and surface roughness for the liquid contact portion as those of the Y-shaped globe valve of the present invention. The connections device may be a flange, welding, grayloc or the like.

[0104] Power for opening and closing the Y-shaped globe valve of the present invention may be human power, air pressure, electricity, hydraulic pressure or any other which can move the stem. When the stem is moved by power, it is desired to incorporate a fail-safe mechanism by which the process becomes safe even when abnormality such as a power failure or disconnection occurs.

[0105] The Y-shaped globe valve of the present invention is installed in the pipe of the polycarbonate production equipment, between a vessel and the pipe, or in a branch of the pipe or the vessel. The Y-shaped globe valve may be used as a sampling valve for sampling the reaction mixture in the pipe by taking it out to the outside of the pipe, or in conjunction with a valve of another type. The Y-shaped globe valve is not limited to examples shown in Figs. 6, 7 and 8 but it is needless to say that it is what is generally used as a Y-shaped globe valve. For example, as shown in Fig. 9, there is a Y-shaped globe valve at a location where the flow of the reaction mixture in a direction perpendicular to the plane of the sheet changes its direction to a downward direction and the liquid flows obliquely downward thereafter. Numerals in Fig. 9 denote the same elements as those of Fig. 7 and Fig. 8.

[0106] The material used for the Y-shaped globe valve of the present invention is preferably subjected to a heat treatment when the influence of residual stress upon the material at high temperatures is taken into consideration.

[0107] When the reaction mixture is not allowed to be leaked to the outside of the Y-shaped globe valve or when gas outside the Y-shaped globe valve is not allowed to enter the inside of the valve, bellows are used to seal a gland portion of the valve.

[0108] When the polycarbonate is not produced, a washing liquid, compressed air or vacuum air is supplied in place of the reaction mixture at the time of washing, start up or shut down the production of the polycarbonate, or inspecting the equipment. It has been confirmed that there is no problem with the function of the valve of the present invention in these cases.

[0109] It is considered that in the present invention, corrosion resistance becomes a problem for the reasons of not only the bad influence of the dissolved ma-

terial such as the prevention of a reaction or the coloration of the polycarbonate but also the formation of a roughened surface probably by corrosion.

[0110] In concrete terms, excellent effects are provided when the material having corrosion resistance is stainless steel and the liquid contact portion is subjected to mechanical finishing such as buffering or to chemical finishing such as electrolytic polishing.

5 10 (2) gear pump:

[0111] The pump used in the production process of a polycarbonate is used at high temperatures under high vacuum. Since the viscosity of the reaction mixture rises to an extremely high level, generally 200 Pa·S to 1,000 Pa·S, which differs according to reaction conditions, along with the proceeding of the above reaction (reaching completion), when the reaction mixture is transferred by a pipe, the pressure of the pump must be raised in accordance with resistance (pressure loss) generated in the pipe. For this purpose, a gear pump has been generally used in the prior art.

[0112] However, as the gear pump of the prior art is made from tool steel having small coefficient of thermal expansion and excellent abrasion resistance, it is inferior in corrosion resistance against the raw materials of the reaction mixture, the material of the gear pump dissolves, the coloration of the reaction mixture occurs, and the dissolved material hinders the activity of a catalyst

20 25 30

and reduces the reaction rate.

[0113] Since the reaction mixture as a product is used for the purpose of lubricating the gear, the reaction mixture which is exposed to high temperatures by heat generated by the gear and high shear for a long time deteriorates and returns into the product, thereby causing deterioration such as coloration, crosslinking or gelation. Thus, a non-negligible influence is exerted upon product quality.

[0114] Further, a residence portion is formed between 40 a bushing which is the shaft end portion of the gear and the bearing of the gear and a cover plate (front plate and back plate). The additionally while section of the pipe at the inlet portion of the reaction mixture is generally circular, an upper portion of the gear generally has a rectangular shape, consequently the sectional shape of the inlet portion of the reaction mixture must be sharply changed to connect these sections having different shapes. Therefore, the residence portion is formed due to the difficulty of machining. Since the sealing of a fixed portion (not driven portion) is metal-to-metal sealing, a residence portion is formed when the gear pump is badly assembled.

[0115] The present inventors have conducted studies on the structure and properties of a gear pump for improving the problem of the prior art and efficiently obtaining a high-quality polymer which rarely produces a deteriorated product in the industrial production process of a polycarbonate. As a result, it has been found that a

high-quality polycarbonate which rarely produces a deteriorated product of a polymer can be obtained by using the following gear pump.

[0116] That is, according to the present invention, there is provided a process for producing a polycarbonate resin through the melt polycondensation of an aromatic diol compound and a carbonic acid diester compound, wherein (2) gear pumps having the following structure and performance are used in the production process.

(2) gear pump; gear pump which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa, a pressure durability of 0.1 MPa or more and a delivery pressure of 1 MPa or more, whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound, which can discharge a reaction mixture under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less, and which has such a structure that the reaction mixture for lubricating contact portions among a gear, a shaft and other gear pump members is discharged to the outside of the pump without returning into the pump.

[0117] A detailed description is subsequently given of the above gear pump (2).

[0118] Equipment comprising the gear pump of the present invention is not particularly limited and the present invention may be carried out in a batch or continuous system. When the batch system is used, two polymerization reactors are generally connected in series, a agitator equipped with a distillator is used for the first reactor, and a agitator without distillator is used for the second reactor to carry out a reaction under different conditions. In this case, it is preferred that both reactors be connected by a pipe equipped with a valve, equipment comprising a gear pump for transferring a reaction mixture as required be used to transfer the reaction product of the first reactor to the second reactor without exposing the reaction mixture to the outside air, and a reaction be carried out to a desired degree of polymerization in the second reactor.

[0119] When the continuous system is used, two or more reactors are generally connected in series, adjacent reactors are connected by a pipe equipped with a valve, equipment comprising a gear pump for transferring a reaction mixture as required is used to supply raw materials and a catalyst to the first reactor continuously while the reactors are maintained under different conditions, and a polycarbonate having a desired degree of polymerization is discharged continuously from the final reactor.

[0120] In the above equipment, a gear pump is required for the supply of the reaction mixture, and the gear pump installed in a vessel or pipe for this purpose plays an important role.

[0121] As described above, the polycarbonate is colored or produces a deteriorated product called

"three-dimensionally crosslinked gel" when it receives long-time heat history though the influence of oxygen is completely eliminated. This reduces the quality of the obtained polycarbonate and causes a serious problem in such application that requires high quality, for example, optical application.

[0122] To solve this problem, it is important to eliminate a residence portion called "dead space" in the equipment. Various studies have been made to eliminate the dead space in the reactor and various proposals have been made. However, the quality of a polycarbonate obtained by melt polymerization is still unsatisfactory in fact.

[0123] The present inventors have conducted intensive studies to solve this problem, have found that a gear pump to which importance was not attached before has a great influence upon the dead space of the whole equipment and have succeeded in greatly improving the quality of a polycarbonate obtained by melt polymerization by using a gear pump having a specific structure.

[0124] In the present invention, a pump for transferring the reaction mixture is such as shown in Fig. 10.

[0125] In the gear pump of the present invention, the term "heat durability" as used herein means that the air-tightness and pressure durability of the gear pump at high temperatures are higher than required air tightness and pressure durability during operation and any problem does not arise in its use even when the gear pump receives force such as the thermal expansion of its material.

When a polycarbonate is produced, the heat resistance temperature of the equipment is preferably set to a range of 150 to 350°C according to its operation conditions. When the heat resistance temperature is higher than 350°C, the material is limited, its processing becomes difficult, the obtained gear pump becomes expensive, a wide clearance must be formed between the parts of the gear pump to eliminate the influence of the thermal expansion of the material constituting the gear pump, and the residence and deterioration of the reaction mixture in this portion readily occur disadvantageously. Since there is a case where a bolt for tightening the parts constituting the gear pump is loosened by thermal expansion, tightening force is preferably increased according to use temperature.

[0126] In the gear pump of the present invention, the expression "substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less" means that the leakage of gas from the outside of the gear pump is 120×10^{-6} MPa·l/h or less based on 1 liter of the inner capacity when the ultimate vacuum degree of the gear pump is 13.3×10^{-6} MPa. With this method for measuring the leakage of gas, the measurement may not be easy because the inner capacity of the gear pump is too small. In this case, when a helium leak test is carried out

at 0.133×10^{-6} MPa, 3×10^{-5} ACC/S or less can be used instead. The measurement method is as follows.

a. gas leakage measurement method

[0127] The measurement of the leakage of gas is carried out by sucking gas inside the gear pump by a vacuum pump to reduce the pressure inside the gear pump to 13.3×10^{-6} MPa, stopping suction and confirming after one hour that the pressure inside the gear pump is lower than a predetermined pressure. When the capacity of the gear pump is 1 liter, for example, the gear pump stands the test if the pressure inside the gear pump is lower than 133×10^{-6} MPa one hour after the suction of the vacuum pump is stopped.

b. helium leak test

[0128] Gas inside the gear pump is sucked by a vacuum pump to reduce the pressure inside the gear pump to 0.133×10^{-6} MPa or less, a helium leak detector is connected to the gear pump and the vacuum pump, the gear pump is covered with a bag while suction by the vacuum pump is continued, helium is blown into the inside of the bag from the outside, and the leakage of helium into the inside of the gear pump is measured by the helium leak detector. If the leakage of the detected helium is 3×10^{-5} ACC/S or less, the gear pump stands the test. The DLMS-TP3E of Nippon Shinku Gijutsu Co., Ltd. may be used as the helium leak detector.

[0129] ACC/S means atm-cc/sec which indicates a volume at 1 atm. and normal temperature as the leakage of helium per unit sec.

[0130] To prevent the gear pump from being exposed to oxygen in the air substantially, the outside of the gear pump is preferably placed in 98 vol% or more of an nitrogen gas atmosphere. Stated more specifically, this is often carried out by blowing from the outside nitrogen gas into a portion where the interior portion and the exterior portion of the gear pump are separated from each other by a sealing portion, such as a drive shaft seal part and a joint between a front plate and a gear case.

[0131] In the gear pump of the present invention, the expression "pressure durability of 0.1 MPa or more" means that the gear pump does not leak a liquid or gas to the outside thereof and does not cause any problem at all in its use even when a predetermined pressure of 0.1 MPa or more is applied to the inside of the gear pump. The pressure durability is generally in the range of 0.1 to 30 MPa.

[0132] In the gear pump of the present invention, the expression "liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound" means that any material is acceptable as the material of the liquid contact portion if it does not impair the function of the gear pump and does not substantially dissolve into the product during the use of the gear pump. The material is desirably stainless steel specified by JIS such as SUS304, SUS304L, SUS316, SUS316L,

SUS630, SCS13, SCS14, SCS16, SCS19, SUS440C, SUS420J2 or SCS2, alloy tool steel specified by JIS such as SKS or SKD, high-speed tool steel such as SKH, nitride steel, plating such as Cr or Ni, stellite face finished or carbon steel lined by a hot isotactic press (HIP) method. Out of these, stainless steel is particularly preferred. The gear, shaft, bushing and body may be made from the same material or different materials but they are preferably made from the same material so as to maintain a clearance between parts formed by thermal expansion at a fixed value from the viewpoints of the stability of the flow rate of a liquid, high delivery pressure and a reduction in dead space.

[0133] In the present invention, the material used for the gear pump is preferably subjected to a heat treatment when the influence of residual stress at high temperatures is taken into consideration.

[0134] Preferably, the gear pump of the present invention is substantially pulseless. The expression "substantially pulseless" means that a change in the flow rate of the reaction mixture discharged by the gear pump is ± 5 % or less, preferably ± 1 % or less, more preferably ± 0.5 % or less of a planned predetermined flow rate.

[0135] In the gear pump of the present invention, the term "delivery pressure" means the pressure of the reaction mixture discharged by the gear pump and this pressure is limited by the viscosity of the reaction mixture used. This is because, when the viscosity is low, the delivery pressure becomes high and the reaction mixture flows backward to the inlet side from the outlet side of the gear pump through a clearance between the gear and the gear, a clearance between the gear and the bushing and a clearance between the gear and the body (that is, a back flow).

[0136] Generally speaking, when the viscosity is 0.01 to 0.1 Pa·S, the delivery pressure is limited to about 1 MPa, when the viscosity is 0.1 to 1 Pa·S, the delivery pressure is limited to about 7 MPa and when the viscosity is more than 1 Pa·S, the delivery pressure is limited to about 7 to 30 MPa.

[0137] In the gear pump of the present invention, the expression "can discharge the reaction mixture under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less" means that the gear pump can transfer the reaction mixture pulseless at a predetermined flow rate and a predetermined delivery pressure even when the pressure at the inlet side of the gear pump is $40,000 \times 10^{-6}$ MPa or less. Generally speaking, this is an important function in the production of a polycarbonate where a reaction is carried out under vacuum.

[0138] In the gear pump of the present invention, the expression "liquid contact portion" means an inside portion contacting the reaction mixture of the gear pump. The portion includes not only a portion which directly contacts the flow of the reaction mixture but also a portion which the reaction mixture used for lubrication contacts or the reaction mixture may enter.

[0139] In the gear pump of the present invention, the

expression "the surface roughness of the liquid contact portion is 10 μm or less" means that the surface roughness is 10 μm or less in terms of R_{max} . The surface roughness is preferably 3.3 μm or less in terms of R_{max} . Specifying a finishing method, the liquid contact portion is finished with the same finishing method illustrated in the above item of (1) value.

[0140] A groove is formed in the bushing of the gear pump of the present invention for the lubrication of the gear so that the reaction mixture on the outlet side is caused to flow between the gear and the bushing and between the shaft and the bushing for lubrication. Since the reaction mixture used for lubrication is deteriorated by the reception of high temperatures and high shearing force in many cases, it is important to discharge this reaction mixture to the outside as much as possible and not to return it into the flow of a product.

[0141] It is preferred to heat the gear pump uniformly with a heating medium jacket, electric heater or the like. In the case of a heating medium jacket, to integrate the heating medium jacket with the gear pump is more preferred than to cover the gear pump with the heating medium jacket from the viewpoint of uniform heating. When the gear pump is relatively large in size, it is preferred to provide a baffle plate in the heating medium jacket or to increase the number of the flow passages of a heating medium so as to uniformly heat the gear pump. When a flange is used to connect the gear pump, it is desired to provide a jacket for circulating the heating medium in the flange.

[0142] Any means may be used to connect the gear pump of the present invention to the pipe or the like if it has the same pressure durability, corrosion resistance and surface roughness for the liquid contact portion as those of the gear pump. For example, it is a flange, welding, grayloc or the like.

[0143] An electric motor is generally used to drive the gear pump of the present invention. A flexible joint is generally used to connect a drive source to the gear pump and desirably can absorb the eccentricity of a drive shaft caused by the thermal expansion of the gear pump or the like.

[0144] An upper limit of the current value of the motor or torque upper limit value is set to protect the gear pump when a metal piece bites the gear during the operation of the gear pump or when abnormal torque is applied to the gear by the deterioration or solidification of the reaction mixture in contact portions among the gear, the shaft and other gear pump members. When the current value of the motor reaches the upper limit, the motor is stopped, or a shear pin or torque limiter is used to disconnect the drive source from the gear pump at a value larger than a predetermined torque value.

[0145] The shaft seal of gear pump of the present invention is used to prevent the reaction mixture inside the gear pump excluding the reaction mixture used for lubrication from contacting the outside when the drive source for driving the gear and shaft is connected to the

gear pump. Any shaft seal is acceptable if it has this function during operation, as exemplified by a mechanical seal, gland packing, sealing coupler, metal seal, ring seal, labyrinth seal, viscous seal and the like. The shaft seal can be used even when it does not contact the drive source. The connection style of the drive source to the gear pump is not limited to one.

[0146] A scraping blade is preferably provided at a shaft end portion (end portion of the shaft) of the gear pump of the present invention to prevent the reaction mixture from residing in the shaft end portion. The scraping blade may have any shape if it has a function to remove the reaction mixture from the shaft end portion. The scraping blade is, for example, a scraper, propeller, screw or the like. Considering production ease and the work efficiency of washing or the like, a simple shape is preferred.

[0147] To eliminate a gap between the bushing and the cover plate of the gear pump of the present invention, the bushing is preferably fixed to the cover plate (front plate or back plate). Fixing means is not particularly limited but may be a bolt, welding or the like.

[0148] The flow passage of the reaction mixture at the inlet port of the gear pump of the present invention, that is, the flow of the reaction mixture at an inlet port portion of the reaction mixture is preferably smooth to prevent the flow line of the reaction mixture from being disturbed. Particularly preferably, the restriction angle of the flow passage is 45° or less in terms of vertical angle. Preferably, the flow passage of the reaction mixture has substantially no level differences except for a required portion for supplying the fluid. The level difference of the required portion for supplying the fluid is the level difference of gear teeth. The expression "has substantially no level differences" means that an end portion is chamfered 2 mm or less, preferably 0.5 mm or less, more preferably 0.1 mm or less.

[0149] In the gear pump of the present invention, the sealing of the fixed portion is the sealing of a gap between parts excluding the shaft and the gear which are movable portions of the gear pump. For example, a gap between the front plate and the gear case, a gap between the back plate and the gear case, a gap between the bushing and the cover plate, and a gap between the pipe and the gear pump are sealed up. This sealing is metal-to-metal sealing which is effected by surface finishing these parts and making them parallel to each other or sealing with a liquid packing. In this case, the surface finish is preferably 25 μm or less, more preferably 6.3 μm or less in terms of R_{max} . Metal-to-metal sealing is generally used but other sealing such as sealing with a liquid packing may be used.

[0150] Particularly when the gear pump is used under vacuum, as only metal-to-metal sealing is insufficient in many cases, an hollow metal O ring is preferably used.

[0151] However, it has been found that the hollow metal O ring has such an advantage that, when a hollow metal O ring is used as a seal, even if vacuum sealing

and also metal-to-metal sealing are insufficient, the reaction mixture does not leak to the outside of the gear pump from the hollow metal O ring, thereby making it possible to minimize a dead space formed in a portion where the reaction mixture leaks.

[0152] Therefore, in a case other than the case where the pressure inside the gear pump is lower than atmospheric pressure, use of the hollow metal O ring is desired from the viewpoint of minimizing the dead space. For this purpose, the hollow metal O ring is desirably placed near the liquid contact portion of the reaction mixture as much as possible.

[0153] An example of the gear pump of the present invention will be described hereinafter with reference to Fig. 10 and Fig. 11. The example shown in Fig. 10 and Fig. 11 is mere an embodiment of the present invention, and it should be understood that the present invention is not limited by these figures.

[0154] In Fig. 10 and Fig. 11, reference notation 102 denotes a gear case. This gear case 102 is connected to an unshown drive unit which is located on a drive shaft seal 112 side of a driving shaft 106 and a follow shaft 107 (the drive shaft seal side of the driving shaft 106 and the follow shaft 107 is called "drive side" and the opposite side is called "counter-drive side" hereinafter). A front plate 101 is attached to the drive side of the gear case 112 and a back plate 103 is attached to the counter-drive side of the gear case 112 to form a body together with the gear case 102.

[0155] This body is covered with a heating medium jacket 105 therearound. A reaction mixture inlet port 110 is formed at one end of the gear case 102 and a reaction mixture outlet port 111 is formed at the other end of the gear case 102.

[0156] The driving shaft 106 is installed in the gear case 102 supported by a bushing 104 and provided with a gear 108. The driving shaft 106 is connected to the unshown drive unit on the drive side, and the gear 108 and the bushing 104 are sealed with the drive shaft seal 112 and separated from the outside of the gear pump.

[0157] A gear 109 is engaged with the gear 108 (see Fig. 11) and attached to the follow shaft 107 supported by the bushing 104 like the gear 108.

[0158] By the rotation of the driving shaft 106 by the unshown drive unit, the gear 108 turns in a direction "a", the gear 109 engaged with the gear 108 turns in a direction "b", the reaction mixture from the reaction mixture inlet port 110 enters the valley portions of the gears 108 and 109 and rotates together with the gears 108 and 109 while it contacts the inner wall of the gear case 102, is forced out of the valley portions of the gears 108 and 109 when the gears 108 and 109 engage with each other on the reaction mixture outlet port 111 side and is supplied to the reaction mixture outlet port 111.

[0159] Metal-to-metal sealing is effected for a gap between the front plate 101 and the gear case 102 and a gap between the back plate 103 and the gear case 102. When the insides of the reaction mixture inlet port 110

and the reaction mixture outlet port 111 become vacuum, metal-to-metal sealing may be insufficient in preventing leakage to the outside of the gear pump. Therefore, an O ring 113 is used for sealing. The O ring 113 serves as a seal for preventing the reaction mixture from leaking to the outside if metal-to-metal sealing should lose its effect owing to some trouble or the like.

[0160] The bushing 104 has a lubricating groove 117 which serves as the flow passage of the reaction mixture as shown in Fig. 13 so that the reaction mixture itself functions as a lubricant for lubricating sliding surfaces 119 between the bushing 104 and the gear 108 and between the bushing 104 and the gear 109 and interfaces between the bushing 104 and the driving shaft 106 and between the bushing 104 and the follow shaft 107 and then is discharged to the outside of the gear pump through an exhaust groove 118.

[0161] In Fig. 13, the reaction mixture for lubrication spreads to a lubricating reaction mixture inlet port 121 (shown by a slant line), passes through the lubricating groove 117 and flows in a direction "c" shown by an arrow.

[0162] Out of the bushings 104, one installed by the drive shaft seal 112 and the front plate 101 has no exhaust groove 118 and the discharged reaction mixture moves in the direction of the drive shaft seal 112 along the driving shaft 106, passes through a gap between the drive shaft seal 112 and the driving shaft 106, and is discharged to the outside of the gear pump while it seals up the gap where a liquid film is formed.

[0163] The "contact portions among the gear, the shaft and other gear pump members" denote portions shown by bold lines in Fig. 10. The word "contact" as used herein does not always mean direct contact but means that the reaction mixture generally lies in these portions as a lubricant.

[0164] In the gear pump of the present invention, means of discharging the reaction mixture used for lubrication to the outside of the system is not particularly limited. The means may be an exhaust port formed in the front plate or the back plate.

[0165] The bushing 104 is fixed to the front plate 101 and to the back plate 103 by a bushing fixing bolt 115 as shown in Fig. 11 and Fig. 12 and sealed with an O ring 116 to prevent the reaction mixture inside from leaking to the outside of the gear pump or the outside air from entering the gear pump.

[0166] Since the bushing 104 is fixed like this, the clearance between the bushing 104 and the gear 108 and the clearance between the bushing 104 and the gear 109 are maintained at a fixed value, the stability of quantitative supply by the gear pump is improved and the self-cleaning properties of the sliding surfaces 119 between the gear 108 and the bushing 104 and between the gear 109 and the bushing 104 are improved compared with a gear pump in which the bushing 104 is not fixed like this. Therefore, this has a great effect in reducing the dead space.

[0167] A scraper 114 having straight thin protrusions is provided at an end portion on the counter-drive side of the driving shaft 116 and both end portions of the follow shaft 107 as shown in Fig. 10 so that the reaction mixture at the end portions of the shafts is stirred by the rotation of the shafts, is prevented from residing in these end portions and can be discharged to the outside smoothly.

[0168] The front plate 101, the gear case 102 and the back plate 103 are preferably made from stainless steel such as SUS420J2, and the driving shaft 106, the follow shaft 107 and the gears 108 and 109 are preferably made from the same material.

[0169] An advantage obtained by making elements from the same material is that even when the temperature of the gear pump is elevated to the operation temperature, a clearance between them is maintained at the same value as that at room temperature because they have the same coefficient of thermal expansion, thereby making it possible to maintain the clearance in the gear pump at a design value. This can prevent the formation of a dead space caused by an increase in the size of the clearance, an increase in the amount of generated heat due to a reduction in the size of the clearance and the gear pump from losing its function when it contacts another element.

[0170] Reactors are connected by a pipe and the gear pump is installed in the pipe. Not shown in Fig. 10, the reaction mixture inlet port 110 and the pipe are connected by a flange and the reaction mixture outlet port 111 and the pipe are also connected by a flange, and a metal O ring is used for sealing.

[0171] The metal O ring used is preferably surface plated to prevent seizure. It is plated with nickel or silver, for example.

[0172] The flow passage of the reaction mixture at the reaction mixture inlet port 110 is restricted at an vertical angle of 20° and is used to supply the reaction mixture to the gear portion. That is, the restriction angle of the flow passage at the reaction mixture inlet port of the gear pump is 20°.

[0173] The reason why the flow passage must be restricted is that the cross section of a connection pipe is generally circular and the cross section of the reaction mixture inlet port formed by the gear 108, the gear 109 and the gear case 102 of the gear pump is rectangular. This restriction angle is preferably as smooth as possible to prevent the formation of a dead space by disturbing the flow line of the reaction mixture.

[0174] The surface of the flow passage of the reaction mixture, that has a surface roughness of 1.6 μm or less in terms of R_{max} to prevent the formation of a fine dead space on the surface of the liquid contact portion.

[0175] In the gear pump constituted as described above, the reaction mixture is supplied continuously from the reaction mixture inlet port 110 and discharged quantitatively from the reaction mixture outlet port 111 to the outside at a predetermined flow rate and pressure.

[0176] When a polycarbonate resin is not produced, a washing liquid, compressed air or vacuum air is supplied in place of the reaction mixture at the time of washing, start up or shut down the production of the polycarbonate, or inspecting the equipment. It has been confirmed that there is no problem with the function of the gear pump in these cases like the reaction mixture.

10 (3) flange;

[0177] A portion for connecting a pipe with another pipe or a pipe with a vessel used in the production process of a polycarbonate is used at high temperatures under high vacuum, and the viscosity of the reaction mixture rises to an extremely high level, generally 200 to 10,000 Pa·S, which differs according to reaction conditions, along with the proceeding of the above reaction (reaching completion). To transfer the reaction mixture by a pipe, as the pressure in the pipe rises in accordance with resistance (pressure loss) generated in the pipe, a flange must stand a high pressure of 0 to 30 MPa. Therefore, the flange must have pressure durability, sealing durability and heat durability.

[0178] The pipe must be connected or disconnected 25 for maintenance at a construction site and there is a tendency toward an increase in the number of pipe joints for the reason of the limitations of tools or the like so as to finish the inner surface of the pipe to a predetermined surface roughness by polishing.

[0179] For these purposes, a flange specified by JIS B2210-1840 has generally been used heretofore.

[0180] Since a gasket is inserted between conventionally used flanges and tightened by a bolt and a nut to connect the flanges, the centers of the pipes are shifted from each other by the tolerance of the hole of the bolt, whereby a level difference is generated in the flange connection portion, or a gap is formed between the flanges by the gasket, thereby forming a dead space.

[0181] The stability of temperature in the pipe is also 40 an important problem for a polycarbonate product whose quality is easily influenced by temperature variations. Although the pipe is generally heated by a heating medium jacket or electric heater, a reduction in temperature easily occurs in the flange portion due to heat radiation because it has a large area, costs are boosted by heating, and heating is liable to become unsatisfactory.

[0182] It is an object of the present invention to provide 45 a flange which improves the above problems of the prior art and is used in an industrial process for efficiently obtaining a high-quality polycarbonate which rarely produces a deteriorated product.

[0183] According to studies conducted by the present inventors, it has been found that a polycarbonate which rarely produces a deteriorated product of a polymer and has high quality can be obtained by using a flange having the following structure and characteristic properties.

[0184] That is, according to the present invention, there is provided a process for producing a polycarbonate resin through the melt polycondensation of an aromatic diol compound and a carbonic acid diester compound, wherein flanges having the following structure and performance are used in the production process.

(3) flange: flange which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less and a pressure durability of 0.1 MPa or more, whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound, and which has a heating medium jacket therein.

[0185] The flange (3) of the present invention will be described in detail hereinafter.

[0186] Equipment comprising the flange of the present invention is not particularly limited and the present invention may be carried out in a batch or continuous system. When the batch system is used, two reactors are generally connected in series, a stirrer equipped with a distillation column is used for the first reactor, and a stirrer without distillation column is used for the second reactor to carry out a reaction under different conditions. In this case, it is preferred that both reactors be connected by a pipe, equipment comprising a pump for transferring the reaction mixture as required be used to transfer the reaction product of the first reactor to the second reactor without exposing the reaction mixture to the outside air, and a reaction be carried out to a desired degree of polymerization in the second reactor.

[0187] When the continuous system is used, two or more reactors are generally connected in series, adjacent reactors are connected by a pipe, the equipment comprising a pump for transferring the reaction mixture as required is used to supply raw materials and a catalyst to the first reactor continuously while the reactors are maintained under different conditions, and a polycarbonate having a desired degree of polymerization is extracted continuously from the final reactor.

[0188] In the above production process, pipes must be connected to supply the reaction mixture, and flanges installed in vessels and pipes for this purpose play an important role.

[0189] The present inventors have found that a flange which is a pipe joint has a great influence upon the dead space of the whole equipment, the stability of temperature is an important factor for a polycarbonate whose product quality is easily influenced by temperature because crystallization is caused by a drop in temperature or heat deterioration is caused by a rise in temperature, and the flange portion easily experiences a drop in temperature by heat radiation due to its large surface area and has a great influence upon the stability of the temperature of the reaction mixture. The present inventors

have succeeded in greatly improving the quality of a reaction mixture obtained by melt polymerization using a flange having a specific structure.

[0190] In the present invention, a flange attached to a pipe for transferring a reaction mixture has a structure shown in Fig. 14, for example.

[0191] In the flange of the present invention, the term "heat durability" as used herein means that the airtightness and pressure durability of the flange are maintained at required values during operation even at high temperatures and the flange has no problem in its use even when it receives force such as the thermal expansion of the material constituting the pipe.

[0192] When a polycarbonate is produced, the heat durability temperature of the equipment is preferably set to a range of 150 to 350°C according to its operation conditions.

[0193] Since there is a case where a bolt for tightening parts constituting the flange is loosened by thermal expansion, tightening force is preferably increased according to use temperature.

[0194] In the flange of the present invention, the expression "substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less" means that the leakage of gas from the outside of the pipe is 120×10^{-6} MPa·l/h or less based on 1 liter of the inner capacity when the ultimate vacuum degree of the pipe including the flange is 13.3×10^{-6} MPa. With this method for measuring the leakage of gas, the measurement may not be easy because the inner capacity of the pipe including the flange is too small. In this case, when a helium leak test is carried out at 0.133×10^{-6} MPa, 3×10^{-5} ACC/S or less can be used instead.

[0195] The gas leakage measurement method (a) and the helium leak test (b) which have been described for the gear pump can be used as the measurement method.

[0196] An outside air contact portion of the flange is desirably placed in 98 vol% or more of a nitrogen gas atmosphere not to be exposed to oxygen contained in the air substantially.

[0197] In the flange of the present invention, the expression "pressure durability of 0.1 MPa or more" means that the flange does not leak a liquid or gas to the outside of the pipe and does not cause any problem at all in its use even when a predetermined pressure of 0.1 MPa or more is applied to the inside of the pipe. The pressure durability is generally in the range of 0.1 to 30 MPa.

[0198] In the flange of the present invention, the expression "liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound" means that any material is acceptable as the material of the liquid contact portion if it does not impair the function of the pipe and does not substantially dissolve into the product during the use of the flange. The material is

desirably stainless steel specified by JIS such as SUS304, SUS304L, SUS316, SUS316L, SUS630, SCS13, SCS14, SCS16 or SCS19, plating such as Cr or Ni, stellite face finished or carbon steel lined by a hot isotactic press (HIP) method. Out of these, stainless steel is particularly preferred. The pipe and the flange may be made from different materials but they are preferably made from the same material to reduce the influence of thermal expansion from the view point of the strength of the pipe.

[0199] In the flange of the present invention, the material of a gasket used for sealing the flange is preferably softer than the material of the flange body from the view point of sealing properties. Illustrative examples of the material include fluororesin (for example, Teflon), perfluoro elastomer resins (for example, Kalrez of Du Pont Dow Elastomers), Ti, Ni, Ag, Al, Cu, asbestos, SUS304, SUS304L, SUS316, SUS316L, SUS317L, SUS321, SUS347, SUS410 (these SUS are specified by JIS), monel metal, incoloy 800, Pb and the like. When the liquid contact surface has a problem with corrosion resistance, it is preferably plated with a material having high corrosion resistance. The gasket is preferably made from a material different from that of the flange body to prevent seizure. Stainless steel is particularly preferred.

[0200] In the flange of the present invention, the material used for the pipe is preferably subjected to a heat treatment when the influence of residual stress at high temperatures is taken into consideration.

[0201] The flange of the present invention is preferably heated uniformly with a heating medium jacket. In the case of a heating medium jacket, the flange may be covered with the heating medium jacket but is preferably integrated with the heating medium jacket by incorporating the heating medium jacket in the flange from the view point of uniform heating. When the flange is relatively large in size, it is preferred to provide a baffle plate in the heating medium jacket or to increase the number of the flow passages of a heating medium so as to uniformly heat the flange.

[0202] The surface roughness of the liquid contact portion of the flange of the present invention is preferably 10 μm or less. The surface roughness means that the surface roughness of the surface in contact with the reaction mixture of the flange is 10 μm or less in terms of R_{max} .

[0203] The surface roughness is preferably 3.3 μm or less in terms of R_{max} . Specifying a finishing method, the liquid contact portion is finished with the same finishing method illustrated in the above item of (1) value.

[0204] In the flange of the present invention, when the flange is connected to a pipe, they are generally connected by welding. However, a carbide is adhered to the inner surface of the pipe because a welded portion of the pipe is exposed to high temperatures, the welded portion or the inner surface of the pipe near the welded portion becomes uneven, and the center of the pipe is shifted by the influence of thermal stress, whereby level

differences are produced. Therefore, it is preferred to carry out post-processing such as surface finishing by polishing.

[0205] In the present invention, any material may be used for the gasket used between flanges if it has required heat durability, pressure durability and corrosion resistance. When a hollow metal O ring is used by forming a groove, it has such an advantage that contact surfaces of the flanges can be easily made metal-to-metal sealing. The hollow metal O ring has another advantage that there is no dead space formed by the deformation or thickness of the gasket itself.

[0206] Further, since sealing properties are provided to the gasket by tightening a bolt and a nut to deform the gasket, the thickness of the flange is made large to provide tightening strength in many cases. However, since the hollow metal O ring has relatively smaller deformation force than other plate gasket or spiral wound gasket, it has still a further advantage that the thickness of the flange can be made relatively thin. Therefore, the hollow metal O ring is preferably used.

[0207] V/#3640 or V/#3641 of Nippon Balker Co., Ltd. or equivalent thereof is preferably used as the hollow metal O ring. The O ring may not have a circular shape

25 and may have an oval or other shape if it surrounds the flow passage of the reaction mixture without a break.

[0208] In the flange of the present invention, a pin or a groove is preferably provided on the surface of the flange to prevent the shifting of the center of the flange.

30 This groove structure is not particularly limited but it is the most preferred to shape a gasket seat for mounting the gasket like a groove in order to improve the fastening pressure of the gasket side and sealing properties.

[0209] In the flange of the present invention, it is preferred to effect metal-to-metal sealing for a gap between the flanges by surface finishing the flanges to a surface roughness of 25 μm or less, preferably 6.3 μm or less, more preferably 1.6 μm or less in terms of R_{max} by calculating the crush margin of the gasket for the inner surface from the gasket seat and by machining the flanges with a lathe to make them parallel to each other from the view point of eliminating the dead space.

[0210] In the flange of the present invention, when the flange is connected to the pipe, the flow passage of the reaction mixture preferably has a smooth shape which does not disturb the flow line of the reaction mixture. Particularly preferably, the flow passage has the same inner diameter as that of the pipe connected thereto. When they cannot have the same diameter in production, they are connected smoothly at a restriction angle of 20° or less in terms of vertical angle. The restriction angle is preferably 10° or less, more preferably 5° or less, much more preferably 1° or less.

[0211] In the present invention, when the flange is relatively large in size and it is difficult to tighten a plurality of bolts and nuts uniformly, a spacer having such a thickness that the tightening pressure of the gasket becomes appropriate when the bolts and nuts are tightened is in-

serted into the gap between the flanges to limit the tightening distance of a screw portion, thereby making it possible to equalize the fastening of bolts and nuts. The insertion of the spacer can prevent the deformation of the flange caused by tightening the bolts and nuts excessively.

[0212] An example of the flange of the present invention will be described hereinafter with reference to Fig. 14. The example shown in Fig. 14 is a mere embodiment of the present invention, and it should be understood that the present invention is not limited by this figure.

[0213] Fig. 14 shows a pair of flanges. A flange (female) 201, a flange (male) 202 and an unshown pipe are made from SUS316. The flange (female) 201 and the flange (male) 202 are tightened to each other by unshown bolts and nuts with predetermined tightening torque. The flange (female) 201 and the flange (male) 202 incorporate a heating medium jacket 203 and are connected to each other by the unshown pipe so that the heating medium circulates to heat and keep warm the flange (female) 201 and the flange (male) 202.

[0214] The flange (female) 201 and the flange (male) 202 are butt welded to the unshown pipe at a pipe welded portion 204.

[0215] The flow passage of the reaction mixture is finished with a buff of #300 and has a surface roughness of 1.6 μm in terms of Rmax to reduce the resistance, an end portion is chamfered 0.1 mm or less and the number of fine dead spaces on the metal surface of the reaction mixture as much as possible.

[0216] The flange (female) 201 and the flange (male) 202 are connected to each other in such a manner that a groove portion (male) 207 and a groove portion (female) 208 are combined together, and their connected surfaces have a surface roughness of 1.6 μm in terms of Rmax and are made parallel to each other by machining with a lathe or the like so as to effect metal-to-metal sealing for a gap between metal-to-metal sealing surfaces 205.

[0217] Vacuum sealing is effected by deforming an unshown hollow metal O ring inserted into a metal O ring attachment seat 206. The size of the groove is set such that the hollow metal O ring does not project from the groove and is deformed to obtain sealing properties.

[0218] The reaction mixture is sealed and separated from the outside by the metal-to-metal sealing surfaces 205 and the hollow metal O ring inserted into the metal O ring attachment seat 206. Owing to metal-to-metal sealing, there is no dead space in fact.

[0219] An unshown spacer is sandwiched between the flange (female) 201 and the flange (male) 202, and the flange (female) 201 and the flange (male) 202 are tightened to each other so that the distance between the flanges should be fixed, tightened more strongly with predetermined torque by elevating the temperature to use temperature and covered with an unshown heat insulating material.

[0220] When a polycarbonate resin is not produced,

a washing liquid, compressed air or vacuum air is supplied in place of the reaction mixture at the time of washing, start up or shut down the production of the polycarbonate, or inspecting the equipment. It has been confirmed that there is no problem with the function of the flange of the present invention in these cases.

(4) flow passage;

[0221] The flow passage in the present invention refers to a space constituted by (i) pipes, (ii) a reaction mixture introduction passage from the inlet of the valve to the sealing surface forming portion (flow passage cut-off portion) of the valve, (iii) a reaction mixture discharge passage from the sealing surface forming portion of the valve to the outlet of the valve and (iv) connection parts for connecting these (such as a socket) and through which a reaction mixture flows, and (v) contacting portion of reaction mixture as final product (such as strand die).

[0222] In the polycarbonate production process, to obtain a high-quality polycarbonate for optical application, it is important to eliminate a residence portion called "dead space" in the transfer route. Various studies have been made to eliminate the dead space in the reactor and various proposals have been made. However, the quality of a polycarbonate obtained by melt polymerization is still unsatisfactory in fact.

[0223] The present inventors have conducted intensive studies in view of this problem and have found that a restricted and/or expanded portion of the flow passage to which importance has not been attached in the prior art, the curvature of the pipe and the connection of the pipe have a great influence upon the dead space of the whole equipment. They have succeeded in greatly improving the quality of a polycarbonate obtained by melt polymerization by using a reducer having a specific structure in the restricted and/or expanded portion of the flow passage.

[0224] In the present invention, the reducer for the flow passage for supplying the reaction mixture is, for example, a reducer as shown in Fig. 16.

[0225] In the flow passage (4) of the present invention, the expression "restricted and/or expanded portion of the flow passage" means a portion whose cross section is reduced or increased of the inside of a member such as a pipe or valve through which the reaction mixture flows. It means what is generally called "reducer" in the case of an ordinary pipe. The reducer is not limited to one specified by JIS but it means a liquid inlet portion of the gear pump, a restricted portion of a strand die, or a cross section changing portion of the flow passage through which the reaction mixture or the polycarbonate flows, such as a portion of the flow passage when the valve is fully opened, and comprehends what is not generally called "reducer".

[0226] In the flow passage of the present invention, the term "heat durability" means that the airtightness

and pressure durability of the flow passage are higher than required air tightness and pressure durability during operation even at high temperatures and any problem does not arise in its use even when the flow passage receives force such as the thermal expansion of the material constituting the flow passage including the restricted and/or expanded portion. When a polycarbonate is produced, the heat durability temperature of the flow passage is preferably set to a range of 150 to 350°C according to its operation conditions.

[0227] In the flow passage of the present invention, the expression "substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less" means that the leakage of gas from the outside including the restricted and/or expanded portion is 120×10^{-6} MPa·l/h or less based on 1 liter of the inner capacity when the ultimate vacuum degree of the flow passage including the restricted and/or expanded portion is 13.3×10^{-6} MPa. Stated more specifically, the leakage is measured by sealing both end portions of the flow passage including the restricted and/or expanded portion.

[0228] In this case, the gas leakage measurement method (a) and the helium leak test (b) which have been described for the gear pump can be used the measurement method.

[0229] An outside air contact portion of the flow passage is desirably placed in 98 vol% or more of an nitrogen gas atmosphere not to be exposed to oxygen contained in the air substantially. This is particularly desirably carried out on a pipe joint portion of the pipe.

[0230] In the flow passage of the present invention, the expression "pressure durability of 0.1 MPa or more" means that the flow passage does not leak a liquid or gas to the outside of the flow passage and does not cause any problem at all in its use even when a predetermined pressure of 0.1 MPa or more is applied to the inside of the flow passage including the restricted and/or expanded portion. The pressure durability is generally a pressure at the outlet of the pump installed in the flow passage and in the range of 0.1 to 30 MPa.

[0231] In the flow passage of the present invention, the expression "liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound" means that any material is acceptable as the material of the liquid contact portion if it does not impair the function of the flow passage and does not substantially dissolve into the product during the use of the flow passage including the restricted and/or expanded portion. The material is desirably stainless steel specified by JIS such as SUS304, SUS304L, SUS316, SUS316L, SUS630, SCS13, SCS14, SCS16, SCS19, SUS440C, SUS420J2 or SCS2, corrosion resistant metal such as nickel or titanium, alloy such as inconel, nickel-chrome iron or hastelloy, plating such as Cr or Ni, stellite face finished, ceramic coating or carbon steel lined by a hot isotactic press (HIP) method. Out of these,

stainless steel is particularly preferred.

[0232] In the present invention, the expression "liquid contact portion" means a reaction mixture contact portion of the inside of the flow passage including the restricted and/or expanded portion.

[0233] In the flow passage of the present invention, the expression "the surface roughness of the liquid contact portion is 10 µm or less" means that the surface roughness is 10 µm or less in terms of Rmax. The surface roughness is preferably 3.3 µm or less in terms of Rmax. Specifying a finishing method, the liquid contact portion is finished with the same finishing method illustrated in the above item of (1) value.

[0234] It is preferred to heat the flow passage including the restricted and/or expanded portion uniformly with a heating medium jacket, electric heater or the like. In the case of a heating medium jacket, to integrate the heating medium jacket with the flow passage body including the restricted and/or expanded portion is more preferred than to cover the flow passage including the restricted and/or expanded portion with the heating medium jacket from the viewpoint of uniform heating. When the flow passage is relatively large in size or when a change in the inner diameter of the flow passage is large due to the restricted and/or expanded portion, it is preferred to provide a baffle plate in the heating medium jacket or to increase the number of the flow passages of the heating medium so as to uniformly heat the flow passage.

[0235] In the present invention, any means may be used to connect the restricted and/or expanded portion of the flow passage to the pipe or the like if it has the same pressure durability, corrosion resistance and surface roughness for the liquid contact portion as those of the restricted and/or expanded portion. It is, for example, a flange, welding, grayloc, socket or the like. Welding is generally used.

[0236] In the present invention, a preferred feature of a connecting method with a pipe is a method by using a socket.

[0237] An example of the socket of the present invention is shown in Fig. 16.

[0238] Fig. 16 shows a pipe 301 before it is connected to a socket 303 and a pipe 302 connected to the socket 303. The liquid contact portion 305 of the pipes 301 and 302 and the socket 303 are finished to a surface roughness Rmax of 1.6 µm or less.

[0239] The contact surfaces with the socket 303 of the pipes 301 and 302 are finished to a surface roughness Rmax of 1.6 µm or less, and the contact surfaces 307 with the pipes 301 and 302 of the socket 303 are finished to a surface roughness Rmax of 1.6 µm or less as well. These contact surfaces of the pipes 301 and 302 and the socket 303 are machined with a lathe to achieve parallelism therebetween, thereby effecting metal-to-metal sealing for a gap between the contact surfaces 306 and 307 together with the above surface roughnesses.

[0240] After the pipes 301 and 302 are inserted into

the socket 303, a groove portion 308 formed in the welding material 304 is welded.

[0241] Any welding material may be used for welding if it has strength required to connect the pipes to the socket, the same material of pipe is preferred as welding material and TIG welding is more preferred. The welding material for welding the pipes 301 and 302 to the socket 303 and the welding material 304 are SUS316 in Fig. 16.

[0242] Since only the surface portions of the pipe 301 and the pipe 302 (portions shown by slant lines in Fig. 16) are welded, the welding material 304 cannot be exposed to the inside of the pipe and contact the reaction mixture. Therefore, the surface finishing of the welded portion which has been required in the prior art is not necessary.

[0243] The reaction mixture is perfectly separated from the outside by metal-to-metal sealing and welding for a gap between the socket 303 and the pipe 301 and a gap between the socket 303 and the pipe 302. Therefore, the reaction mixture does not enter the gaps formed between these sealed surfaces. Even if it enters the gaps, it is extremely small in quantity and it is almost impossible that the reaction mixture entering the gaps deteriorates and the deteriorated product enters the flow of the reaction mixture by any chance.

[0244] Since the socket is much more compact than other connection means such as a flange and a jacket for heating the pipes can be laid in the socket when double pipe heat tracing are used, heat retaining properties are higher than other pipe joints.

[0245] In the present invention, the flow of the reaction mixture at the inlet portion of the reaction mixture of the restricted and/or expanded portion is desirably smooth to prevent the flow line of the reaction mixture from being disturbed. Particularly preferably, the restriction or expansion angle of the flow passage is 20° or less in terms of vertical angle. It is more preferably 14° or less in terms of vertical angle. The flow passage having substantially no level differences is preferably used. The expression "substantially no level differences" means that the end portion is preferably chamfered 2 mm or less, more preferably 0.5 mm or less, much more preferably 0.1 mm or less.

[0246] The vertical angle in this case is an angle shown by "α" when the flow passage is of a concentric type as shown in Fig. 17 and Fig. 18. When the flow passage is of an eccentric type as shown in Fig. 19 and Fig. 20, the vertical angle is an angle shown by "β".

[0247] In the present invention, when the restricted and/or expanded portion is used in a horizontal flow passage, a flow passage of an eccentric type is often used to prevent the formation of an air pool. In this case, the "horizontal flow passage" refers to a flow passage having an angle of 0 to 45° formed between the flow direction of the reaction mixture in the flow passage and the ground.

[0248] Examples of the restricted and/or expanded portion according to the present invention are shown in

Figs. 17 to 20. The examples shown in Figs. 17 to 20 are mere embodiments of the present invention, and it should be understood that the present invention is not limited by these figures.

5 [0249] Fig. 17 and Fig. 18 show a reducer of a concentric type and Fig. 19 and Fig. 20 shows a reducer of an eccentric type. Fig. 17 and Fig. 18 show a reducer body 401 before it is welded to a pipe. A liquid contact portion 404 has a surface roughness of 1.6 μm in terms of Rmax and butt welded to the pipe at a welded portion 405 on a small pipe side 402 and a large pipe side 403.

10 [0250] Any welding material may be used for the welded portion 405 if it has strength required to connect the pipe to a reducer but TIG welding is preferred. The reducer body 401, the welding material and the pipe are made from SUS316. The welded portion is smoothly finished to a surface roughness of 1.6 μm in terms of Rmax to prevent the formation of a dead space by disturbing the flow line of the reaction mixture.

15 [0251] The restriction or expansion angle is $\alpha = 20^\circ$ for the flow passage of a concentric type shown in Fig. 17 and Fig. 18 and $\beta = 10^\circ$ for the flow passage of an eccentric type shown in Fig. 19 and Fig. 20.

20 [0252] In the pipe including the reducer constituted above, the reaction mixture is continuously transferred.

25 [0253] In the flow passage of the present invention, a curved portion of the pipe for transferring the reaction mixture is formed by bending a pipe shown in Fig. 21, for example, with a bender. The term "pipe" as used herein means a pipe in a general sense but can mean all flow passages through which the reaction mixture passes, such as a lubricating portion of the bearing of the gear pump or a flow passage portion of a strand die except for seat of the value.

30 [0254] In the present invention, the inside of the curved portion preferably has a smooth shape which does not disturb the flow line of the reaction mixture. The curvature of the pipe must be 5 times or more the inner diameter of the pipe, preferably 8 times or more the inner diameter of the pipe.

35 [0255] In the present invention, the angle of the curved portion is not fixed to 45°, 90° or 180° but may be any angle.

40 [0256] In the present invention, a curved portion having substantially no level differences in a welded portion or in the inside of the pipe is also preferably used. The expression "substantially no level differences" means that the end portion is preferably chamfered 2 mm or less, more preferably 0.5 mm or less, much more preferably 0.1 mm or less.

45 [0257] In the present invention, bending is cold bending with a pipe bender, burn bending or high-frequency bending. Cold bending is preferred because it does not cause a change in composition or the formation of a roughened surface by heating.

50 [0258] In the present invention, allowable flatness after bending should be 7.5 % or less of the nominal diameter of the pipe. The cross section of the curved por-

tion is desirably spherical at any location.

[0259] The "allowable flatness" as used herein indicates a percentage (%) obtained by subtracting the length of a short diameter from the length of a long diameter of the cross section of the curved portion and dividing the obtained value by the nominal diameter of the pipe when the cross section of the curved portion changes its shape to an oval shape.

[0260] The nominal diameter of the pipe used herein is specified by JIS G 3452-1988 and expressed as nominal diameter A.

[0261] An example of the curved portion according to the present invention is shown in Fig. 21. The example shown in Fig. 21 is a mere embodiment of the present invention, and it should be understood that the present invention is not limited by this figure.

[0262] In Fig. 21, a curved portion 406 is formed by bending a pipe and has a curvature radius "r" which satisfies $r \geq 5 \times d$ (d is the inner diameter of the pipe). The bending angle is 90°.

[0263] Not shown, both ends of the curved portion are butt welded to pipes.

[0264] A welding material may be preferably used similar material of pipe if it has strength required to connect the pipe, but TIG welding is preferred. The curved portion 406, the welding material and the pipe are preferably made from stainless steel such as SUS316. The welded portion is smoothly finished to a surface roughness of 1.6 μm or less in terms of Rmax to prevent the formation of a dead space by disturbing the flow line of the reaction mixture.

[0265] The reaction mixture is continuously transferred in the pipe including the curved portion thus constituted.

Examples

[0266] The following examples of the present invention are given below. These examples are provided for the purpose of further illustrating the present invention but are in no way to be taken as limiting.

[0267] In the present invention, intrinsic viscosity and Rmax were measured as described in "a" and "b" below. The gas leakage measurement method of the present invention is carried out as described in "c". In the following examples, a helium leak test equivalent to the gas leakage measurement method of the present invention was used.

a. intrinsic viscosity

[0268] The intrinsic viscosity $[\eta]$ was obtained by measuring a methylene chloride solution of the reaction mixture having a concentration of 0.7 g/dl with an Ubbelohde's viscometer.

b. Rmax measurement method

[0269] Rmax indicates surface roughness expressed in pm. Rmax is measured in accordance with JIS B0601.

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c. gas leakage measurement method

[0270] The measurement of the leakage of gas is carried out by sucking gas inside the transfer members by

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a vacuum pump to reduce the pressure inside the transfer members to 13.3×10^{-6} MPa, stopping suction by the vacuum pump and confirming after one hour that the pressure inside the transfer members is lower than a predetermined pressure when the capacity of the transfer members is 1 liter. The transfer members stands the test if the pressure inside the transfer members is lower than 133×10^{-6} MPa one hour after suction by the vacuum pump is stopped.

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Example 1

[0271] Diphenyl carbonate was charged into a dissolution tank equipped with a stirrer in an amount of 1.05 mols based on 1 mol of 2,2-bis(4-hydroxyphenyl)propane, the inside of the tank was substituted with nitrogen, 2,2-bis(4-hydroxyphenyl)propane was dissolved in diphenyl carbonate at 150°C, and the resulting mixture solution was transferred to a raw material storage tank maintained at 150°C.

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[0272] Thereafter, the mixture was continuously supplied to a vertical reactor whose inside temperature was maintained at 240°C and inside pressure at $1,333 \times 10^{-6}$ MPa and which was equipped with a fractionator and agitator at a rate of 60 kg/hr, 1×10^{-6} equivalent of a bisphenol A disodium salt and 1×10^{-4} equivalent of tetramethyl ammonium hydroxide were continuously added to 1 mol of 2,2-bis(4-hydroxyphenyl)propane, and a reaction was carried out by removing the produced phenol from the fractionator. The valve of the present invention shown in Fig. 6 was installed at the raw material inlet and the prepolymer outlet of this reactor. The obtained reaction mixture was continuously discharged using a gear pump. The polymerization degree of the obtained reaction mixture was obtained by measuring intrinsic viscosity thereof. As a result, a reaction mixture having a $[\eta]$ of 0.16 was obtained.

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[0273] Thereafter, the reaction mixture was continuously supplied to a horizontal reactor whose inside temperature was maintained at 270°C and inside pressure at 133×10^{-6} MPa. The reaction mixture was further polymerized while the produced phenol was removed to the outside of the system to continuously produce a polycarbonate having a $[\eta]$ of 0.45. The viscosity of the reaction mixture at the outlet was 1,400 Pa-S at 270°C.

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[0274] A Y-shaped globe valve shown in Fig. 6 was installed at the reaction mixture inlet and the reaction mixture outlet of the horizontal reactor.

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[0275] The specifications of the valve of the present

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[0276] The valve of the present invention has a valve body 100, a valve seat 102, a valve seat 104, a valve seat 106, a valve seat 108, a valve seat 110, a valve seat 112, a valve seat 114, a valve seat 116, a valve seat 118, a valve seat 120, a valve seat 122, a valve seat 124, a valve seat 126, a valve seat 128, a valve seat 130, a valve seat 132, a valve seat 134, a valve seat 136, a valve seat 138, a valve seat 140, a valve seat 142, a valve seat 144, a valve seat 146, a valve seat 148, a valve seat 150, a valve seat 152, a valve seat 154, a valve seat 156, a valve seat 158, a valve seat 160, a valve seat 162, a valve seat 164, a valve seat 166, a valve seat 168, a valve seat 170, a valve seat 172, a valve seat 174, a valve seat 176, a valve seat 178, a valve seat 180, a valve seat 182, a valve seat 184, a valve seat 186, a valve seat 188, a valve seat 190, a valve seat 192, a valve seat 194, a valve seat 196, a valve seat 198, a valve seat 200, a valve seat 202, a valve seat 204, a valve seat 206, a valve seat 208, a valve seat 210, a valve seat 212, a valve seat 214, a valve seat 216, a valve seat 218, a valve seat 220, a valve seat 222, a valve seat 224, a valve seat 226, a valve seat 228, a valve seat 230, a valve seat 232, a valve seat 234, a valve seat 236, a valve seat 238, a valve seat 240, a valve seat 242, a valve seat 244, a valve seat 246, a valve seat 248, a valve seat 250, a valve seat 252, a valve seat 254, a valve seat 256, a valve seat 258, a valve seat 260, a valve seat 262, a valve seat 264, a valve seat 266, a valve seat 268, a valve seat 270, a valve seat 272, a valve seat 274, a valve seat 276, a valve seat 278, a valve seat 280, a valve seat 282, a valve seat 284, a valve seat 286, a valve seat 288, a valve seat 290, a valve seat 292, a valve seat 294, a valve seat 296, a valve seat 298, a valve seat 300, a valve seat 302, a valve seat 304, a valve seat 306, a valve seat 308, a valve seat 310, a valve seat 312, a valve seat 314, a valve seat 316, a valve seat 318, a valve seat 320, a valve seat 322, a valve seat 324, a valve seat 326, a valve seat 328, a valve seat 330, a valve seat 332, a valve seat 334, a valve seat 336, a valve seat 338, a valve seat 340, a valve seat 342, a valve seat 344, a valve seat 346, a valve seat 348, a valve seat 350, a valve seat 352, a valve seat 354, a valve seat 356, a valve seat 358, a valve seat 360, a valve seat 362, a valve seat 364, a valve seat 366, a valve seat 368, a valve seat 370, a valve seat 372, a valve seat 374, a valve seat 376, a valve seat 378, a valve seat 380, a valve seat 382, a valve seat 384, a valve seat 386, a valve seat 388, a valve seat 390, a valve seat 392, a valve seat 394, a valve seat 396, a valve seat 398, a valve seat 400, a valve seat 402, a valve seat 404, a valve seat 406, a valve seat 408, a valve seat 410, a valve seat 412, a valve seat 414, a valve seat 416, a valve seat 418, a valve seat 420, a valve seat 422, a valve seat 424, a valve seat 426, a valve seat 428, a valve seat 430, a valve seat 432, a valve seat 434, a valve seat 436, a valve seat 438, a valve seat 440, a valve seat 442, a valve seat 444, a valve seat 446, a valve seat 448, a valve seat 450, a valve seat 452, a valve seat 454, a valve seat 456, a valve seat 458, a valve seat 460, a valve seat 462, a valve seat 464, a valve seat 466, a valve seat 468, a valve seat 470, a valve seat 472, a valve seat 474, a valve seat 476, a valve seat 478, a valve seat 480, a valve seat 482, a valve seat 484, a valve seat 486, a valve seat 488, a valve seat 490, a valve seat 492, a valve seat 494, a valve seat 496, a valve seat 498, a valve seat 500, a valve seat 502, a valve seat 504, a valve seat 506, a valve seat 508, a valve seat 510, a valve seat 512, a valve seat 514, a valve seat 516, a valve seat 518, a valve seat 520, a valve seat 522, a valve seat 524, a valve seat 526, a valve seat 528, a valve seat 530, a valve seat 532, a valve seat 534, a valve seat 536, a valve seat 538, a valve seat 540, a valve seat 542, a valve seat 544, a valve seat 546, a valve seat 548, a valve seat 550, a valve seat 552, a valve seat 554, a valve seat 556, a valve seat 558, a valve seat 560, a valve seat 562, a valve seat 564, a valve seat 566, a valve seat 568, a valve seat 570, a valve seat 572, a valve seat 574, a valve seat 576, a valve seat 578, a valve seat 580, a valve seat 582, a valve seat 584, a valve seat 586, a valve seat 588, a valve seat 590, a valve seat 592, a valve seat 594, a valve seat 596, a valve seat 598, a valve seat 600, a valve seat 602, a valve seat 604, a valve seat 606, a valve seat 608, a valve seat 610, a valve seat 612, a valve seat 614, a valve seat 616, a valve seat 618, a valve seat 620, a valve seat 622, a valve seat 624, a valve seat 626, a valve seat 628, a valve seat 630, a valve seat 632, a valve seat 634, a valve seat 636, a valve seat 638, a valve seat 640, a valve seat 642, a valve seat 644, a valve seat 646, a valve seat 648, a valve seat 650, a valve seat 652, a valve seat 654, a valve seat 656, a valve seat 658, a valve seat 660, a valve seat 662, a valve seat 664, a valve seat 666, a valve seat 668, a valve seat 670, a valve seat 672, a valve seat 674, a valve seat 676, a valve seat 678, a valve seat 680, a valve seat 682, a valve seat 684, a valve seat 686, a valve seat 688, a valve seat 690, a valve seat 692, a valve seat 694, a valve seat 696, a valve seat 698, a valve seat 700, a valve seat 702, a valve seat 704, a valve seat 706, a valve seat 708, a valve seat 710, a valve seat 712, a valve seat 714, a valve seat 716, a valve seat 718, a valve seat 720, a valve seat 722, a valve seat 724, a valve seat 726, a valve seat 728, a valve seat 730, a valve seat 732, a valve seat 734, a valve seat 736, a valve seat 738, a valve seat 740, a valve seat 742, a valve seat 744, a valve seat 746, a valve seat 748, a valve seat 750, a valve seat 752, a valve seat 754, a valve seat 756, a valve seat 758, a valve seat 760, a valve seat 762, a valve seat 764, a valve seat 766, a valve seat 768, a valve seat 770, a valve seat 772, a valve seat 774, a valve seat 776, a valve seat 778, a valve seat 780, a valve seat 782, a valve seat 784, a valve seat 786, a valve seat 788, a valve seat 790, a valve seat 792, a valve seat 794, a valve seat 796, a valve seat 798, a valve seat 800, a valve seat 802, a valve seat 804, a valve seat 806, a valve seat 808, a valve seat 810, a valve seat 812, a valve seat 814, a valve seat 816, a valve seat 818, a valve seat 820, a valve seat 822, a valve seat 824, a valve seat 826, a valve seat 828, a valve seat 830, a valve seat 832, a valve seat 834, a valve seat 836, a valve seat 838, a valve seat 840, a valve seat 842, a valve seat 844, a valve seat 846, a valve seat 848, a valve seat 850, a valve seat 852, a valve seat 854, a valve seat 856, a valve seat 858, a valve seat 860, a valve seat 862, a valve seat 864, a valve seat 866, a valve seat 868, a valve seat 870, a valve seat 872, a valve seat 874, a valve seat 876, a valve seat 878, a valve seat 880, a valve seat 882, a valve seat 884, a valve seat 886, a valve seat 888, a valve seat 890, a valve seat 892, a valve seat 894, a valve seat 896, a valve seat 898, a valve seat 900, a valve seat 902, a valve seat 904, a valve seat 906, a valve seat 908, a valve seat 910, a valve seat 912, a valve seat 914, a valve seat 916, a valve seat 918, a valve seat 920, a valve seat 922, a valve seat 924, a valve seat 926, a valve seat 928, a valve seat 930, a valve seat 932, a valve seat 934, a valve seat 936, a valve seat 938, a valve seat 940, a valve seat 942, a valve seat 944, a valve seat 946, a valve seat 948, a valve seat 950, a valve seat 952, a valve seat 954, a valve seat 956, a valve seat 958, a valve seat 960, a valve seat 962, a valve seat 964, a valve seat 966, a valve seat 968, a valve seat 970, a valve seat 972, a valve seat 974, a valve seat 976, a valve seat 978, a valve seat 980, a valve seat 982, a valve seat 984, a valve seat 986, a valve seat 988, a valve seat 990, a valve seat 992, a valve seat 994, a valve seat 996, a valve seat 998, a valve seat 1000, a valve seat 1002, a valve seat 1004, a valve seat 1006, a valve seat 1008, a valve seat 1010, a valve seat 1012, a valve seat 1014, a valve seat 1016, a valve seat 1018, a valve seat 1020, a valve seat 1022, a valve seat 1024, a valve seat 1026, a valve seat 1028, a valve seat 1030, a valve seat 1032, a valve seat 1034, a valve seat 1036, a valve seat 1038, a valve seat 1040, a valve seat 1042, a valve seat 1044, a valve seat 1046, a valve seat 1048, a valve seat 1050, a valve seat 1052, a valve seat 1054, a valve seat 1056, a valve seat 1058, a valve seat 1060, a valve seat 1062, a valve seat 1064, a valve seat 1066, a valve seat 1068, a valve seat 1070, a valve seat 1072, a valve seat 1074, a valve seat 1076, a valve seat 1078, a valve seat 1080, a valve seat 1082, a valve seat 1084, a valve seat 1086, a valve seat 1088, a valve seat 1090, a valve seat 1092, a valve seat 1094, a valve seat 1096, a valve seat 1098, a valve seat 1100, a valve seat 1102, a valve seat 1104, a valve seat 1106, a valve seat 1108, a valve seat 1110, a valve seat 1112, a valve seat 1114, a valve seat 1116, a valve seat 1118, a valve seat 1120, a valve seat 1122, a valve seat 1124, a valve seat 1126, a valve seat 1128, a valve seat 1130, a valve seat 1132, a valve seat 1134, a valve seat 1136, a valve seat 1138, a valve seat 1140, a valve seat 1142, a valve seat 1144, a valve seat 1146, a valve seat 1148, a valve seat 1150, a valve seat 1152, a valve seat 1154, a valve seat 1156, a valve seat 1158, a valve seat 1160, a valve seat 1162, a valve seat 1164, a valve seat 1166, a valve seat 1168, a valve seat 1170, a valve seat 1172, a valve seat 1174, a valve seat 1176, a valve seat 1178, a valve seat 1180, a valve seat 1182, a valve seat 1184, a valve seat 1186, a valve seat 1188, a valve seat 1190, a valve seat 1192, a valve seat 1194, a valve seat 1196, a valve seat 1198, a valve seat 1200, a valve seat 1202, a valve seat 1204, a valve seat 1206, a valve seat 1208, a valve seat 1210, a valve seat 1212, a valve seat 1214, a valve seat 1216, a valve seat 1218, a valve seat 1220, a valve seat 1222, a valve seat 1224, a valve seat 1226, a valve seat 1228, a valve seat 1230, a valve seat 1232, a valve seat 1234, a valve seat 1236, a valve seat 1238, a valve seat 1240, a valve seat 1242, a valve seat 1244, a valve seat 1246, a valve seat 1248, a valve seat 1250, a valve seat 1252, a valve seat 1254, a valve seat 1256, a valve seat 1258, a valve seat 1260, a valve seat 1262, a valve seat 1264, a valve seat 1266, a valve seat 1268, a valve seat 1270, a valve seat 1272, a valve seat 1274, a valve seat 1276, a valve seat 1278, a valve seat 1280, a valve seat 1282, a valve seat 1284, a valve seat 1286, a valve seat 1288, a valve seat 1290, a valve seat 1292, a valve seat 1294, a valve seat 1296, a valve seat 1298, a valve seat 1300, a valve seat 1302, a valve seat 1304, a valve seat 1306, a valve seat 1308, a valve seat 1310, a valve seat 1312, a valve seat 1314, a valve seat 1316, a valve seat 1318, a valve seat 1320, a valve seat 1322, a valve seat 1324, a valve seat 1326, a valve seat 1328, a valve seat 1330, a valve seat 1332, a valve seat 1334, a valve seat 1336, a valve seat 1338, a valve seat 1340, a valve seat 1342, a valve seat 1344, a valve seat 1346, a valve seat 1348, a valve seat 1350, a valve seat 1352, a valve seat 1354, a valve seat 1356, a valve seat 1358, a valve seat 1360, a valve seat 1362, a valve seat 1364, a valve seat 1366, a valve seat 1368, a valve seat 1370, a valve seat 1372, a valve seat 1374, a valve seat 1376, a valve seat 1378, a valve seat 1380, a valve seat 1382, a valve seat 1384, a valve seat 1386, a valve seat 1388, a valve seat 1390, a valve seat 1392, a valve seat 1394, a valve seat 1396, a valve seat 1398, a valve seat 1400, a valve seat 1402, a valve seat 1404, a valve seat 1406, a valve seat 1408, a valve seat 1410, a valve seat 1412, a valve seat 1414, a valve seat 1416, a valve seat 1418, a valve seat 1420, a valve seat 1422, a valve seat 1424, a valve seat 1426, a valve seat 1428, a valve seat 1430, a valve seat 1432, a valve seat 1434, a valve seat 1436, a valve seat 1438, a valve seat 1440, a valve seat 1442, a valve seat 1444, a valve seat 1446, a valve seat 1448, a valve seat 1450, a valve seat 1452, a valve seat 1454, a valve seat 1456, a valve seat 1458, a valve seat 1460, a valve seat 1462, a valve seat 1464, a valve seat 1466, a valve seat 1468, a valve seat 1470, a valve seat 1472, a valve seat 1474, a valve seat 1476, a valve seat 1478, a valve seat 1480, a valve seat 1482, a valve seat 1484, a valve seat 1486, a valve seat 1488, a valve seat 1490, a valve seat 1492, a valve seat 1494, a valve seat 1496, a valve seat 1498, a valve seat 1500, a valve seat 1502, a valve seat 1504, a valve seat 1506, a valve seat 1508, a valve seat 1510, a valve seat 1512, a valve seat 1514, a valve seat 1516, a valve seat 1518, a valve seat 1520, a valve seat 1522, a valve seat 1524, a valve seat 1526, a valve seat 1528, a valve seat 1530, a valve seat 1532, a valve seat 1534, a valve seat 1536, a valve seat 1538, a valve seat 1540, a valve seat 1542, a valve seat 1544, a valve seat 1546, a valve seat 1548, a valve seat 1550, a valve seat 1552, a valve seat 1554, a valve seat 1556, a valve seat 1558, a valve seat 1560, a valve seat 1562, a valve seat 1564, a valve seat 1566, a valve seat 1568, a valve seat 1570, a valve seat 1572, a valve seat 1574, a valve seat 1576, a valve seat 1578, a valve seat 1580, a valve seat 1582, a valve seat 1584, a valve seat 1586, a valve seat 1588, a valve seat 1590, a valve seat 1592, a valve seat 1594, a valve seat 1596, a valve seat 1598, a valve seat 1600, a valve seat 1602, a valve seat 1604, a valve seat 1606, a valve seat 1608, a valve seat 1610, a valve seat 1612, a valve seat 1614, a valve seat 1616, a valve seat 1618, a valve seat 1620, a valve seat 1622, a valve seat 1624, a valve seat 1626, a valve seat 1628, a valve seat 1630, a valve seat 1632, a valve seat 1634, a valve seat 1636, a valve seat 1638, a valve seat 1640, a valve seat 1642, a valve seat 1644, a valve seat 1646, a valve seat 1648, a valve seat 1650, a valve seat 1652, a valve seat 1654, a valve seat 1656, a valve seat 1658, a valve seat 1660, a valve seat 1662, a valve seat 1664, a valve seat 1666, a valve seat 1668, a valve seat 1670, a valve seat 1672, a valve seat 1674, a valve seat 1676, a valve seat 1678, a valve seat 1680, a valve seat 1682, a valve seat 1684, a valve seat 1686, a valve seat 1688, a valve seat 1690, a valve seat 1692, a valve seat 1694, a valve seat 1696, a valve seat 1698, a valve seat 1700, a valve seat 1702, a valve seat 1704, a valve seat 1706, a valve seat 1708, a valve seat 1710, a valve seat 1712, a valve seat 1714, a valve seat 1716, a valve seat 1718, a valve seat 1720, a valve seat 1722, a valve seat 1724, a valve seat 1726, a valve seat 1728, a valve seat 1730, a valve seat 1732, a valve seat 1734, a valve seat 1736, a valve seat 1738, a valve seat 1740, a valve seat 1742, a valve seat 1744, a valve seat 1746, a valve seat 1748, a valve seat 1750, a valve seat 1752, a valve seat 1754, a valve seat 1756, a valve seat 1758, a valve seat 1760, a valve seat 1762, a valve seat 1764, a valve seat 1766, a valve seat 1768, a valve seat 1770, a valve seat 1772, a valve seat 1774, a valve seat 1776, a valve seat 1778, a valve seat 1780, a valve seat 1782, a valve seat 1784, a valve seat 1786, a valve seat 1788, a valve seat 1790, a valve seat 1792, a valve seat 1794, a valve seat 1796, a valve seat 1798, a valve seat 1800, a valve seat 1802, a valve seat 1804, a valve seat 1806, a valve seat 1808, a valve seat 1810, a valve seat 1812, a valve seat 1814, a valve seat 1816, a valve seat 1818, a valve seat 1820, a valve seat 1822, a valve seat 1824, a valve seat 1826, a valve seat 1828, a valve seat 1830, a valve seat 1832, a valve seat 1834, a valve seat 1836, a valve seat 1838, a valve seat 1840, a valve seat 1842, a valve seat 1844, a valve seat 1846, a valve seat 1848, a valve seat 1850, a valve seat 1852, a valve seat 1854, a valve seat 1856, a valve seat 1858, a valve seat 1860, a valve seat 1862, a valve seat 1864, a valve seat 1866, a valve seat 1868, a valve seat 1870, a valve seat 1872, a valve seat 1874, a valve seat 1876, a valve seat 1878, a valve seat 1880, a valve seat 1882, a valve seat 1884, a valve seat 1886, a valve seat 1888, a valve seat 1890, a valve seat 1892, a valve seat 1894, a valve seat 1896, a valve seat 1898, a valve seat 1900, a valve seat 1902, a valve seat 1904, a valve seat 1906, a valve seat 1908, a valve seat 1910, a valve seat 1912, a valve seat 1914, a valve seat 1916, a valve seat 1918, a valve seat 1920, a valve seat 1922, a valve seat 1924, a valve seat 1926, a valve seat 1928, a valve seat 1930, a valve seat 1932, a valve seat 1934, a valve seat 1936, a valve seat 1938, a valve seat 1940, a valve seat 1942, a valve seat 1944, a valve seat 1946, a valve seat 1948, a valve seat 1950, a valve seat 1952, a valve seat 1954, a valve seat 1956, a valve seat 1958, a valve seat 1960, a valve seat 1962, a valve seat 1964, a valve seat 1966, a valve seat 1968, a valve seat 1970, a valve seat 1972, a valve seat 1974, a valve seat 1976, a valve seat 1978, a valve seat 1980, a valve seat 1982, a valve seat 1984, a valve seat 1986, a valve seat 1988, a valve seat 1990, a valve seat 1992, a valve seat 1994, a valve seat 1996, a valve seat 1998, a valve seat 2000, a valve seat 2002, a valve seat 2004, a valve seat 2006, a valve seat 2008, a valve seat 2010, a valve seat 2012, a valve seat 2014, a valve seat 2016, a valve seat 2018, a valve seat 2020, a valve seat 2022, a valve seat 2024, a valve seat 2026, a valve seat 2028, a valve seat 2030, a valve seat 2032, a valve seat 2034, a valve seat 2036, a valve seat 2038, a valve seat 2040, a valve seat 2042, a valve seat 2044, a valve seat 2046, a valve seat 2048, a valve seat 2050, a valve seat 2052, a valve seat 2054, a valve seat 2056, a valve seat 2058, a valve seat 2060, a valve seat 2062, a valve seat 2064, a valve seat 2066, a valve seat 2068, a valve seat 2070, a valve seat 2072, a valve seat 2074, a valve seat 2076, a valve seat 2078, a valve seat 2080, a valve seat 2082, a valve seat 2084, a valve seat 2086, a valve seat 2088, a valve seat 2090, a valve seat 2092, a valve seat 2094, a valve seat 2096, a valve seat 2098, a valve seat 2100, a valve seat 2102, a valve seat 2104, a valve seat 2106, a valve seat 2108, a valve seat 2110, a valve seat 2112, a valve seat 2114, a valve seat 2116, a valve seat 2118, a valve seat 2120, a valve seat 2122, a valve seat 2124, a valve seat 2126, a valve seat 2128, a valve seat 2130, a valve seat 2132, a valve seat 2134, a valve seat 2136, a valve seat 2138, a valve seat 2140, a valve seat 2142, a valve seat 2144, a valve seat 2146, a valve seat 2148, a valve seat 2150, a valve seat 2152, a valve seat 2154, a valve seat 2156, a valve seat 2158, a valve seat 2160, a valve seat 2162

invention used in this example were as follows.

[0276] The valve body was made from SCS14, stem from SUS316, disk from SUS316, seal from STELLITE FACE#6, and gland packing from T/#9075. The liquid contact portion of the valve had a pressure durability of 15 MPa, a heat durability of 300°C and an ultimate vacuum of 1.33×10^{-6} MPa (3×10^{-5} ACC/S or less according to a helium leak test). When the surface of the liquid contact portion was subjected to mechanical finishing (buff of #300), it had a surface roughness Rmax of 2.4 μm . A heating medium jacket was incorporated in the valve body to uniformly heat the valve. A disk was provided at the end of the stem. Flanges were used to connect the valve to a pipe.

[0277] When the reaction mixture was sampled at the outlet of the horizontal reactor after 16 days of continuous operation, dissolved in 1 kg of a methylene chloride solution and filtered with a 30 μm -mesh filter, about 65 foreign substances (in average) such as gels or thermally deteriorated products of the polycarbonate based on 1 kg of the polycarbonate were detected on the filter. There was a tendency toward an increase in the number of foreign substances as the operation time became longer.

Example 2

[0278] A polymerization reaction was carried out in the same manner as in Example 1 except that a Y-shaped globe valve shown in Fig. 8 was installed as the valve of the present invention.

[0279] The specifications of the valve were as follows.

[0280] The valve body was made from SCS14, stem from SUS316, seal from STELLITE FACE#6 and gland packing from T/#9075. An O ring of Kalrez was attached to the stem, and the liquid contact portion had a pressure durability of 15 MPa, a heat durability of 300°C and an ultimate vacuum of 1.33×10^{-6} MPa (3×10^{-5} ACC/S or less according to a helium leak test). When the surface of the liquid contact portion was subjected to mechanical finishing (buff of #300), it had a surface roughness Rmax of 2.4 μm . A heating medium jacket was incorporated in the valve body to uniformly heat the valve. A disk was not provided at the end of the stem, the diameter of the stem was made equal to that of a pipe, and the stem was welded to a pipe. A spacer having a smooth curved surface that does not disturb the flow of a polycarbonate resin was installed at the end of the stem.

[0281] When the reaction mixture was sampled at the outlet of the horizontal reactor after 16 days of operation, dissolved in 1 kg of methylene chloride and filtered with a 30 μm -mesh filter, about 3 foreign substances (in average) were existent on the filter based on 1 kg of the polycarbonate.

[0282] When the inner wall of the valve stem was observed with naked eye after 16 days of operation, it retained the same metal gloss as that before operation.

Example 3

[0283] A polymerization reaction was carried out in the same manner as in Example 1 except that an O ring of Kalrez was not attached to the stem of the same Y-shaped globe valve as in Example 2 installed as the valve of the present invention.

[0284] When the reaction mixture was sampled at the outlet of a horizontal reactor after 16 days of operation, dissolved in 1 kg of methylene chloride and filtered with a 30 μm -mesh filter, about 15 foreign substances (in average) were existent on the filter based on 1 kg of the polycarbonate.

Example 4

[0285] A polymerization reaction was carried out in the same manner as in Example 1 except that a spacer was not provided at the stem of the same Y-shaped globe valve as in Example 3 installed as the valve of the present invention.

[0286] When the reaction mixture was sampled at the outlet of a horizontal reactor after 16 days of operation, dissolved in 1 kg of methylene chloride and filtered with a 30 μm -mesh filter, about 25 foreign substances (in average) were existent on the filter based on 1 kg of the polycarbonate.

Comparative Example 1

[0287] A polymerization reaction was carried out in the same manner as in Example 1 except that a plug valve shown in Fig. 4 was used in place of the valve of the present invention.

[0288] The specifications of the valve were as follows.

[0289] The valve body was made from SCS14, plug from SUS316 and gland packing from T/#9075. The liquid contact portion had a pressure durability of 15 MPa and a heat durability of 300°C. The sliding portion of the valve was coated with Triballoy #400. When the surface of the liquid contact portion was finished with a buff of #100, it had a surface roughness Rmax of 15 μm .

[0290] When the reaction mixture was sampled at the outlet of a horizontal reactor after 16 days of operation, dissolved in 1 kg of methylene chloride and filtered with a 30 μm -mesh filter, about 100 foreign substances were existent on the filter based on 1 kg of the polycarbonate. There was a tendency toward an increase in the number of foreign substances as the operation time became longer.

[0291] Right after the valve was operated, countless foreign substances were existent on the filter. This phenomenon was not observed in any of the above Examples 1 to 4.

[0292] When the surface of the plug of the valve was observed with the naked eye after 16 days of operation, brown substances were adhered to the surface of the plug.

Comparative Example 2

[0293] A polymerization reaction was carried out in the same manner as in Example 1 except that a T-shaped globe valve shown in Fig. 5 was used in place of the valve of the present invention.

[0294] The specifications of the valve were as follows.

[0295] The valve body was made from SCS14, stem from SUS316, disk from SUS316, seat from STELLITE FACE#6 and gland packing from T/#9075. The liquid contact portion had a pressure durability of 15 MPa, a heat durability of 300°C and an ultimate vacuum degree of 1.33×10^{-6} MPa (3×10^{-5} ACC/S or less according to a helium leak test). When the surface of the liquid contact portion was finished with a buff of #100, it had a surface roughness Rmax of 15 µm. A heating medium jacket was incorporated in the valve body to uniformly heat the valve, and flanges were used to connect the valve to a pipe.

[0296] When the reaction mixture was sampled at the outlet of a horizontal reactor after 16 days of operation, dissolved in 1 kg of methylene chloride and filtered with a 30 µm-mesh filter, about 100 foreign substances were existent on the filter based on 1 kg of the polycarbonate. There was a tendency toward an increase in the number of foreign substances as the operation time became longer.

[0297] Right after the valve was operated, countless foreign substances were existent on the filter. This phenomenon was not observed in any of the above Examples 1 to 4.

[0298] When the rear surface of the disk of the valve was observed with the naked eye after 16 days of operation, brown substances were adhered to the surface.

Example 5

[0299] Diphenyl carbonate was charged into a dissolution tank equipped with a stirrer in an amount of 1.05 mols based on 1 mol of 2,2-bis(4-hydroxyphenyl)propane, the inside of the tank was substituted with nitrogen, 2,2-bis(4-hydroxyphenyl)propane was dissolved in diphenyl carbonate at 150°C, and the resulting mixture solution was transferred to a raw material storage tank maintained at 150°C.

[0300] Thereafter, the mixture was continuously supplied to a vertical reactor whose inside temperature was maintained at 240°C and inside pressure at $1,333 \times 10^{-6}$ MPa and which was equipped with a fractionator and agitator at a rate of 60 kg/hr, 1×10^{-6} equivalent of a bisphenol A disodium salt and 1×10^{-4} equivalent of tetramethyl ammonium hydroxide were continuously added to 1 mol of 2,2-bis(4-hydroxyphenyl)propane, and a reaction was carried out by removing the produced phenol from the fractionator.

[0301] The obtained reaction mixture was continuously discharged using the gear pump of the present invention (to be referred to as "former gear pump"). The

polymerization degree of the obtained reaction mixture was obtained by measuring intrinsic viscosity thereof. As a result, a reaction mixture having a $[\eta]$ of 0.16 was obtained.

5 [0302] Thereafter, the reaction mixture was continuously supplied to a horizontal reactor whose inside temperature was maintained at 270°C and inside pressure at 133×10^{-6} MPa. The reaction mixture was further polymerized while the produced phenol was removed to 10 the outside of the system to continuously produce a polycarbonate having a $[\eta]$ of 0.35. The gear pump of the present invention (to be referred to as "latter gear pump") was used to discharge the obtained reaction mixture continuously. The viscosity of the reaction mixture at the outlet was 300 Pa·S at 270°C.

15 [0303] As for the gear pump of the present invention, the front plate, gear case and back plate were made from SUS420J2, the gear, bushing and shaft were made from SUS420J2 and had a surface roughness Rmax of 20 1.6 µm by buffing, and a hollow metal O ring was used to seal the fixed portion. The reaction mixture used to lubricate contact portions among the gear, shaft and other gear pump members was discharged to the outside of the pump without returning into the reaction mixture 25 as final product, the flow passage of the reaction mixture had a restriction angle of 20° in terms of vertical angle, a gland packing was used as a shaft seal for the driving shaft of the former gear pump and a labyrinth seal was used as a shaft seal for the driving shaft of the latter gear pump.

30 [0304] Further, the size of the flow passage and the driving power of the gear pump were adjusted according to the viscosity of the reaction mixture. The size of the flow passage was adjusted because a pressure loss in 35 the flow passage of the reaction mixture has a proportional relationship with viscosity ("the flow passage of the reaction mixture" is an item by which the diameters of the inlet and the outlet of the gear pump, the size of the lubricating groove in the gear and the clearance of 40 the drive shaft seal were adjusted). The reason why the driving force of the gear pump was adjusted was that the required driving force of the gear pump was increased by a rise in the viscosity of the reaction mixture.

[0305] When 1 kg of the polymer obtained at the outlet 45 of the horizontal reactor was sampled after 10 days of operation, dissolved in methylene chloride and filtered with a 30 µm-mesh filter, about 8 foreign substances (in average) were detected on the filter based on 1 kg of the polycarbonate. The expression "foreign substances" as used herein means a gel, highly crystallized product and thermally deteriorated product of a polycarbonate.

[0306] When the gear pump was disassembled after 55 40 days of operation, a deteriorated reaction mixture was adhered between the cover plate and the bushing and to end portions of the driving shaft and the follow shaft. The other liquid contact portions of the reaction mixture retained almost the same metal gloss as that

before operation.

Example 6

[0307] A polymerization reaction was carried out in the same manner as in Example 5 except that the bushing for supporting the shaft of the gear was fixed to the cover plate, a gap between the cover plate and the bushing was substantially eliminated, and a scraper was installed at end portions of the driving shaft and the follow shaft.

[0308] When 1 kg of the polymer obtained at the outlet of a horizontal reactor after 10 days of operation, dissolved in methylene chloride and filtered with a 30 μm mesh filter, about 2 foreign substances (in average) were detected on the filter based on 1 kg of the polycarbonate.

[0309] When the gear pump was disassembled after 40 days of operation, the liquid contact portion of the reaction mixture retained almost the same metal gloss as that before operation.

Comparative Example 3

[0310] A polymerization reaction was carried out in the same manner as in Example 5 except that the gear pump used was such that the front plate, gear case and back plate were made from SUS316, a reaction mixture used to lubricate contact portions among the gear, shaft and other gear pump members returned into the reaction mixture as final product after lubrication, the bushing for supporting the shaft of the gear was not fixed to the cover plate, there was no scraper at the end portion of the shaft, the restriction angle of the flow passage of the reaction mixture was 60° in terms of vertical angle, a mechanical seal was used for the former gear pump as a drive shaft seal, there was a level difference in the flow passage at the inlet of the reaction mixture formed by chamfering 4 mm, the surface roughness of the liquid contact portion was set to R_{max} of 12.5 μm , and the sealing of the fixed portion was metal-to-metal sealing.

[0311] When 1 kg of the polymer obtained at the outlet of a horizontal reactor was sampled after 10 days of operation, dissolved in methylene chloride and filtered with a 30 μm filter, about 90 foreign substances (in average) were detected on the filter based on 1 kg of the polycarbonate. There was a tendency toward an increase in the number of foreign substances as the operation time became longer.

[0312] When the gear pump was disassembled after 40 days of operation, a reaction mixture which was discolored in the flow passage of the reaction mixture used to lubricate contact portions among the gear, shaft and other gear pump members was adhered to end portions of the gear shaft, and a discolored reaction mixture resided in the gap between the cover plate and the bushing. The shaft end portion showed the marks of a deteriorated reaction mixture adhered thereto. The mechan-

ical seal as a drive shaft seal for the former gear pump showed the marks of a sealing solution leaking inside. A discolored reaction mixture was also adhered to a level difference portion of the flow passage of the reaction mixture and small recessed portions in the flow passage of the polycarbonate.

Comparative Example 4

[0313] A polymerization reaction was carried out in the same manner as in Example 5 except that the gear pump used was made from SKD11, the reaction mixture used to lubricate contact portions among the gear, shaft and other gear pump members returned into the reaction mixture as final product again after lubrication, the bushing for supporting the shaft of the gear was not fixed to the cover plate, there was no scraper at the end portion of the shaft, the restriction angle of the flow passage of the reaction mixture was 60° in terms of vertical angle, the drive shaft seal was a gland packing, there was a level difference formed by chamfering 4 mm in the flow passage at the inlet of the reaction mixture, the surface roughness of the liquid contact portion was set to 12.5 μm , the sealing of the fixed portion was metal-to-metal sealing.

[0314] When 1 kg of the polymer obtained at the outlet of a horizontal reactor was sampled after 10 days of operation, dissolved in methylene chloride and filtered with a 30 μm filter, about 200 foreign substances were detected on the filter based on 1 kg of the polycarbonate. When the obtained reaction mixture was observed with the naked eye, it assumed a brown color.

[0315] When the gear pump was disassembled after 40 days of operation, a discolored reaction mixture was adhered to the whole flow passage of the reaction mixture. A discolored reaction mixture was adhered to the inside of the flow passage of the reaction mixture used to lubricate contact portions among the gear, shaft and other gear pump members, and the discolored reaction mixture resided in the gap between the cover plate and the bushing. The shaft end portion showed the marks of the deteriorated reaction mixture adhered thereto.

Example 7

[0316] Diphenyl carbonate was charged into a dissolution tank equipped with a stirrer in an amount of 1.05 mols based on 1 mol of 2,2-bis(4-hydroxyphenyl)propane, the inside of the tank was substituted with nitrogen, 2,2-bis(4-hydroxyphenyl)propane was dissolved in diphenyl carbonate at 150°C, and the resulting mixture solution was transferred to a raw material storage tank maintained at 150°C.

[0317] Thereafter, the mixture was continuously supplied to a vertical reactor whose inside temperature was maintained at 240°C and inside pressure at $1,333 \times 10^{-6}$ MPa and which was equipped with a fractionator and agitator by a pump connected with flanges at a rate of

60 kg/hr, 1×10^{-6} equivalent of a bisphenol A disodium salt and 1×10^{-4} equivalent of tetramethyl ammonium hydroxide were continuously added to 1 mol of 2,2-bis(4-hydroxyphenyl)propane, and a reaction was carried out by removing the produced phenol from the fractionator. The obtained reaction mixture was continuously discharged using a gear pump from the pipe connected with the flanges of the present invention. The polymerization degree of the obtained reaction mixture was obtained by measuring intrinsic viscosity thereof. As a result, a reaction mixture having a $[\eta]$ of 0.16 was obtained.

[0318] Thereafter, the reaction mixture was continuously supplied to a horizontal reactor whose inside temperature was maintained at 270°C and inside pressure at 133×10^{-6} MPa.

[0319] The reaction mixture was further polymerized while the produced phenol was removed to the outside of the system to continuously produce a polycarbonate having a $[\eta]$ of 0.35.

[0320] The flange for the object of this example was used to connect connection pipes for the horizontal reactor, the connection pipes for the horizontal reactor to the horizontal reactor, the horizontal reactor to the outlet pipe thereof, and the outlet pipes for the horizontal reactor.

[0321] The viscosity of the reaction mixture on the outlet side was 300 Pa·s.

[0322] The flange used to attain the object of this example was a flange incorporating a heating medium jacket as shown in Fig. 15 and made from SUS316, the reaction mixture contact surface and the pipe and socket metal-to-metal sealing surfaces thereof had a surface roughness of 1.6 μm in terms of R_{max} , and TIG welding was used. A hollow metal O ring prepared by plating SUS316 with silver was used for a gasket and the gasket was tightened with predetermined torque and tightened more strongly by elevating temperature.

[0323] This flange has a heat durability of 300°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less and a pressure durability of 15 MPa.

[0324] The flange shown in Fig. 15 differs from the flange shown in Fig. 14 in that there is no groove portion (male) 207 and no groove portion (female) 208 and the heating medium jacket shown in Fig. 14 does not extend to the collar portion of the flange.

[0325] When 1 kg of the polymer obtained at the outlet of the horizontal reactor was sampled after 10 days of operation, dissolved in methylene chloride and filtered with a 30 μm filter, about 15 foreign substances (in average) were detected on the filter based on 1 kg of the polycarbonate. The expression "foreign substances" as used herein means a gel, highly crystallized product and thermally deteriorated product of a polycarbonate.

[0326] When the flange was disassembled after 30 days of operation, the reaction mixture contact portion had the same metal gloss as that before operation and

a slight amount of a deteriorated product of the polycarbonate resin was adhered to the metal-to-metal sealing surface.

5 Example 8

[0327] A polymerization reaction was carried out in the same manner as in Example 7 except that the flange shown in Fig. 14 was used in place of the flange shown in Fig. 15.

[0328] When 1 kg of the polymer obtained at the outlet of the horizontal reactor was sampled after 10 days of operation, dissolved in methylene chloride and filtered with a 30 μm filter, about 2 foreign substances (in average) were detected on the filter based on 1 kg of the polycarbonate.

[0329] When the flange was disassembled after 30 days of operation, the reaction mixture contact portion had the same metal gloss as that before operation and the metal-to-metal sealing surface had the same metal gloss as that before operation as well.

Comparative Example 5

25 [0330] A polymerization reaction was carried out in the same manner as in Example 7 except that a flange of the same type as that shown in Fig. 15 which had no metal O ring attachment portion and no heating medium jacket was used in place of the flange shown in Fig. 15 and an asbestos spiral wound gasket provided with inner and outer rings was used.

[0331] When 1 kg of the polymer obtained at the outlet of the horizontal reactor was sampled after 10 days of operation, dissolved in methylene chloride and filtered with a 30 μm filter, about 55 almost white foreign substances (in average) were detected on the filter based on 1 kg of the polycarbonate.

[0332] When the flange was disassembled after 16 days of operation, white foreign substances were adhered to the reaction mixture contact portion in large quantities and a deteriorated product of the polycarbonate resin was adhered to the metal-to-metal sealing surface.

45 Example 9

[0333] Diphenyl carbonate was charged into a dissolution tank equipped with a stirrer in an amount of 1.05 mols based on 1 mol of 2,2-bis(4-hydroxyphenyl)propane, the inside of the tank was substituted with nitrogen, 2,2-bis(4-hydroxyphenyl)propane was dissolved in diphenyl carbonate at 150°C, and the resulting mixture solution was transferred to a raw material storage tank maintained at 150°C.

[0334] Thereafter, the mixture was continuously supplied to a vertical reactor whose inside temperature was maintained at 240°C and inside pressure at $1,333 \times 10^{-6}$ MPa and which was equipped with a fractionator and

agitator at a rate of 60 kg/hr, 1×10^{-6} equivalent of a bisphenol A disodium salt and 1×10^{-4} equivalent of tetramethyl ammonium hydroxide were continuously added to 1 mol of 2,2-bis(4-hydroxyphenyl)propane, and a reaction was carried out by removing the produced phenol from the fractionator.

[0335] The obtained reaction mixture was continuously discharged using a gear pump. The polymerization degree of the obtained reaction mixture was obtained by measuring intrinsic viscosity thereof. As a result, a reaction mixture having a $[\eta]$ of 0.16 was obtained.

[0336] Thereafter, the reaction mixture was continuously supplied to a horizontal reactor whose inside temperature was maintained at 270°C and inside pressure at 133×10^{-6} MPa. The reaction mixture was further polymerized while the produced phenol was removed to the outside of the system to continuously produce a polycarbonate having a $[\eta]$ of 0.35.

[0337] The obtained reaction mixture was continuously discharged using a gear pump. The viscosity of the reaction mixture at the outlet was 300 Pa·S at 270°C.

[0338] The reducer of the present invention was used in all the restricted and expanded portions of the pipes used to connect the dissolution tank to the vertical reactor, gear pump, horizontal reactor and gear pump for the discharge of a reaction mixture and all the branches of the pipes.

[0339] The used pipes and the reducer of the present invention were made from SUS316 and had a surface roughness of 1.6 μm in terms of Rmax by buffing, and a reducer of an eccentric type having a restriction or expansion angle of 10° in terms of vertical angle was connected to the pipe by TIG welding in such a manner that an upper portion of the reducer became horizontal.

[0340] When 1 kg of the polymer obtained at the outlet of the horizontal reactor was sampled after 10 days of operation, dissolved in methylene chloride and filtered with a 30 μm -mesh filter, about 2 foreign substances (in average) were detected on the filter based on 1 kg of the polycarbonate. The expression "foreign substances" as used herein means a gel, highly crystallized product and thermally deteriorated product of a polycarbonate.

[0341] When the liquid contact portion of the reducer was observed through a fiberscope after 40 days of operation, it retained almost the same metal gloss as that before operation. However, when the restricted and expanded portions of the liquid contact portions of the gear pump and the valve used were dissembled and inspected, a trace amount of a brown product was adhered.

Example 10

[0342] A polymerization reaction was carried out in the same manner as in Example 9 except that the restriction and expansion of the liquid contact portions of the gear pump and the valve used were set to a vertical

angle of 20°.

[0343] When 1 kg of the polymer obtained at the outlet of the horizontal reactor was sampled after 10 days of operation, dissolved in methylene chloride and filtered with a 30 pm-mesh filter, about 8 foreign substances (in average) were detected on the filter based on 1 kg of the polycarbonate.

[0344] When the liquid contact surfaces of the gear pump and the valve were observed through a fiberscope after 40 days of operation, they retained almost the same metal gloss as that before operation.

Comparative Example 6

[0345] A polymerization reaction was carried out in the same manner as in Example 9 except that a reducer of a concentric type having a restriction and expansion angle of 45° in terms of vertical angle was used.

[0346] When 1 kg of the polymer obtained at the outlet of the horizontal reactor was sampled after 10 days of operation, dissolved in methylene chloride and filtered with a 30 μm -mesh filter, about 15 foreign substances (in average) were detected on the filter based on 1 kg of the polycarbonate. There was a tendency toward an increase in the number of the foreign substances as the operation time became longer.

[0347] When the reducer was observed through a fiberscope after 40 days of operation, a slight amount of a brown product was adhered to the liquid contact surface of the reducer. A larger amount of the brown product was adhered to an upper portion of the liquid contact surface of the reducer than a lower portion of the liquid contact surface, and a larger amount of the brown product was adhered to the expanded portion than the restricted portion.

Example 11

[0348] Diphenyl carbonate was charged into a dissolution tank equipped with a stirrer in an amount of 1.05 mols based on 1 mol of 2,2-bis(4-hydroxyphenyl)propane, the inside of the tank was substituted with nitrogen, 2,2-bis(4-hydroxyphenyl)propane was dissolved in diphenyl carbonate at 150°C, and the resulting mixture solution was transferred to a raw material storage tank maintained at 150°C.

[0349] Thereafter, the mixture was continuously supplied to a vertical reactor whose inside temperature was maintained at 240°C and inside pressure at $1,333 \times 10^{-6}$ MPa and which was equipped with a fractionator and agitator at a rate of 60 kg/hr, 1×10^{-6} equivalent of a bisphenol A disodium salt and 1×10^{-4} equivalent of tetramethyl ammonium hydroxide were continuously added to 1 mol of 2,2-bis(4-hydroxyphenyl)propane, and a reaction was carried out by removing the produced phenol from the fractionator.

[0350] The obtained reaction mixture was continuously discharged using a gear pump. The polymeriza-

tion degree of the obtained reaction mixture was obtained by measuring intrinsic viscosity thereof. As a result, a reaction mixture having a $[\eta]$ of 0.16 was obtained.

[0351] Thereafter, the reaction mixture was continuously supplied to a horizontal reactor whose inside temperature was maintained at 270°C and inside pressure at 133×10^{-6} MPa. The reaction mixture was further polymerized while the produced phenol was removed to the outside of the system to continuously produce a polycarbonate having a $[\eta]$ of 0.35. The viscosity of the reaction mixture at the outlet was 300 Pa·S at 270°C.

[0352] The pipe and curved portion of the present invention used were cold bent with a bender, made from SUS316 and finished by fluid polishing and buffering to a surface roughness of 1.6 μm in terms of Rmax. The curvature of the pipe was 9 times the inner diameter of the pipe. The pipe was connected by TIG welding.

[0353] In this example, the curved portion of the present invention was used for all the curved portions of pipes used to connect the dissolution tank to the vertical reactor, gear pump, horizontal reactor and gear pump for the extraction of a polycarbonate and in all branches of these pipes for the transfer of the reaction mixture.

[0354] When 1 kg of the polymer obtained at the outlet of the horizontal reactor was sampled after 10 days of operation, dissolved in methylene chloride and filtered with a 30 μm -mesh filter, about 2 foreign substances (in average) were detected on the filter based on 1 kg of the polycarbonate. The expression "foreign substances" as used herein means a gel, highly crystallized product and thermally deteriorated product of a polycarbonate.

[0355] When the inside of the pipe was observed through a fiberscope after 40 days of operation, the reaction mixture contact portion retained almost the same metal gloss as that before operation.

Comparative Example 7

[0356] A polymerization reaction was carried out in the same manner as in Example 11 except that an elbow pipe specified by JIS B 2312-1986 was used as a pipe having a curved portion.

[0357] When 1 kg of the polymer obtained at the outlet of the horizontal reactor was sampled after 10 days of operation, dissolved in methylene chloride and filtered with a 30 μm -mesh filter, about 15 foreign substances (in average) were detected on the filter based on 1 kg of the polycarbonate. There was a tendency toward an increase in the number of the foreign substances as the operation time became longer.

[0358] When the inside of the elbow was observed through a fiberscope after 40 days of operation, a slight amount of a brown product was adhered to the liquid contact surface of the elbow.

Example 12

[0359] Diphenyl carbonate was charged into a dissolution tank equipped with a stirrer in an amount of 1.05 mols based on 1 mol of 2,2-bis(4-hydroxyphenyl)propane, the inside of the tank was substituted with nitrogen, 2,2-bis(4-hydroxyphenyl)propane was dissolved in diphenyl carbonate at 150°C, and the resulting mixture solution was transferred to a raw material storage tank maintained at 150°C.

[0360] Thereafter, the mixture was continuously supplied to a vertical reactor whose inside temperature was maintained at 240°C and inside pressure at $1,333 \times 10^{-6}$ MPa and which was equipped with a fractionator and agitator at a rate of 60 kg/hr, 1 $\times 10^{-6}$ equivalent of a bisphenol A disodium salt and 1 $\times 10^{-4}$ equivalent of tetramethyl ammonium hydroxide were continuously added to 1 mol of 2,2-bis(4-hydroxyphenyl)propane, and a reaction was carried out by removing the produced phenol from the fractionator.

[0361] The obtained reaction product was continuously discharged using a gear pump. The polymerization degree of the obtained reaction product was obtained by measuring intrinsic viscosity thereof. As a result, a reaction mixture having a $[\eta]$ of 0.16 was obtained.

[0362] Thereafter, the reaction mixture was continuously supplied to a horizontal reactor whose inside temperature was maintained at 270°C and inside pressure at 133×10^{-6} MPa. The reaction mixture was further polymerized while the produced phenol was removed to the outside of the system to continuously produce a polycarbonate having a $[\eta]$ of 0.35. The obtained reaction mixture was continuously discharged using a gear pump and the reducer of the present invention. The viscosity of the reaction mixture at the outlet was 300 Pa·S at 270°C.

[0363] In this example, the flange, reducer, bender bent pipe, valve and gear pump of the present invention were used for all the flanges, the restricted, expanded and curved portions of the pipes and the valves as transfer members and branch pipes provided for these transfer members installed in a production line from the dissolution tank, vertical reactor, gear pump, horizontal reactor and gear pump for the discharge of a polycarbonate as shown in Fig. 1.

[0364] The transfer members of the present invention have a heat durability of 300°C, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less and a pressure resistance of 15 MPa.

[0365] As for the materials of the liquid contact portions of the transfer members used, the gear pump was made from SUS420J2 and other members were made from SUS316. A hollow metal O ring prepared by plating SUS316 with silver was used for a gasket and the gasket was tightened with predetermined torque and tightened more strongly by elevating temperature.

[0366] All the transfer members of the present inven-

tion were kept warm by a heating medium jacket to prevent the temperature of the reaction mixture from being lowered than the temperature of each reactor. The liquid contact portions had a surface roughness of 1.6 μm in terms of R_{max} by fluid polishing or buffing and were made smooth with substantially no level differences, and the metal-touch surface of each of the transfer members had a surface roughness of 1.6 μm in terms of R_{max} . TIG welding was used for welded portions.

[0367] All the flow passages of the present invention except the inlet portion of the gear pump which had a restriction or expansion angle of 20° in terms of vertical angle had a vertical angle of 10° and were cold bent to ensure that the curvature became 9 times the inner diameter of the flow passage.

[0368] The reducer used in the pipe portions was an eccentric type reducer shown in Fig. 20 which was installed in such a manner that an upper portion thereof became horizontal.

[0369] The flange of the present invention incorporated a heating medium jacket as shown in Fig. 14.

[0370] The gear pump of the present invention discharged the reaction mixture used to lubricate contact portions among the gear, shaft and other gear pump members to the outside of the pump so that it did not return into the reaction mixture as a final product again. A gland packing was used as a seal for the drive shaft of the former gear pump and a labyrinth seal was used as a seal for the driving shaft of the latter gear pump.

[0371] The delivery pressure of the former gear pump was adjusted to 1 MPa and the delivery pressure of the latter gear pump was adjusted to 3 MPa.

[0372] The bushing for supporting the shaft of the gear was fixed to the cover plate, a gap between the cover plate and the bushing was substantially eliminated, and a scraper was installed at end portions of the driving shaft and the follow shaft.

[0373] The valve of the present invention is a Y-shaped globe valve shown in Fig. 8, the diameter of the stem was made substantially equal to the inner diameter of the pipe, and a spacer having a smooth curved surface that did not disturb the flow of the reaction mixture was installed at an end portion of the stem. The seat was made from STELLITE FACE #6.

[0374] When 1 kg of the polymer was sampled at the outlet of the horizontal reactor after 10 days of operation, dissolved in methylene chloride and filtered with a 30 μm -mesh filter, about 1 foreign substances (in average) were detected on the filter based on 1 kg of the polycarbonate. The expression "foreign substances" as used herein means a gel, highly crystallized product and thermally deteriorated product of a polycarbonate. When the number of foreign substances were counted likewise for 90 days of operation, it remained the same at about 1 based on 1 kg of the polycarbonate. When the mixture of foreign substances at the time of sampling was taken into consideration, it was estimated that there was substantially no foreign substance which was formed during

the production of the polycarbonate.

[0375] When the transfer members were observed through a fiberscope after 90 days of operation, the liquid contact portions of the reaction mixture maintained almost the same metal gloss as that before operation.

Example 13

[0376] A polymerization reaction was carried out in 10 the same manner as in Example 12 except that a flange shown in Fig. 15 which had no metal O ring attachment portion and no heating medium jacket was used in place of the flange used in a production line from the former gear pump to the polycarbonate discharge portion and 15 an asbestos spiral wound gasket provided with inner and outer rings was used.

[0377] When 1 kg of the polymer was sampled at the 20 outlet of the horizontal reactor after 10 days of operation, dissolved in methylene chloride and filtered with a 30 μm -mesh filter, 5 to 10 foreign substances were detected on the filter based on 1 kg of the polycarbonate.

[0378] When the transfer members were observed 25 through a fiberscope after 16 days of operation, the liquid contact portions of the reaction mixture excluding the flange portions maintained almost the same metal gloss as that before operation. White foreign matter was adhered to the liquid contact portions of the flange portions in large quantities and a brown deteriorated product was adhered to the gasket between the flanges.

Example 14

[0379] A polymerization reaction was carried out in 35 the same manner as in Example 12 except that a gear pump in which the front plate, gear case and back plate were made from SUS316, the reaction mixture used to lubricate contact portions among the gear, shaft and other gear pump members returned into the reaction mixture which would become a product after lubrication 40 again, the bushing for supporting the shaft of the gear was not fixed to the cover plate, there was no scraper at end portions of the shafts, the restriction angle of the flow passage at the inlet of the gear pump was 60° in terms of vertical angle, a mechanical seal was used for the former gear pump, the liquid contact portions had a surface roughness of 12.5 μm in terms of R_{max} , and the sealing of the fixed portion was metal-to-metal sealing was used in place of the gear pump of the present invention.

[0380] When 1 kg of the polymer was sampled at the 45 outlet of the horizontal reactor after 10 days of operation, dissolved in methylene chloride and filtered with a 30 μm -mesh filter, about 40 foreign substances (in average) were detected on the filter based on 1 kg of the polycarbonate.

[0381] When the transfer members were observed 50 through a fiberscope after 16 days of operation, the liquid contact portions of the reaction mixture excluding the

gear pump portions maintained almost the same metal gloss as that before operation. When the gear pump was disassembled and inspected, a discolored reaction mixture used to lubricate contact portions among the gear, shaft and other gear pump members was adhered to the inside of the flow passages, and a brown deteriorated product was adhered between the cover plate and the bushing. The shaft end portion showed the marks of a brown product adhered thereto. The mechanical seal as a drive shaft seal for the former gear pump showed the marks of a sealing solution leaking inside and mixed into the reaction mixture.

Example 15

[0382] A polymerization reaction was carried out in the same manner as in Example 12 except that a T-shaped globe valve shown in Fig. 5 was used as a valve between a vertical reactor and a horizontal reactor in place of the valve of the present invention.

[0383] When 1 kg of the polymer was sampled at the outlet of the horizontal reactor after 10 days of operation, dissolved in methylene chloride and filtered with a 30 μm -mesh filter, about 50 foreign substances (in average) were detected on the filter based on 1 kg of the polycarbonate. Countless foreign substances were existent on the filter right after the valve was operated.

[0384] A required amount of the reaction mixture could not be supplied unless the delivery pressure of the former gear pump was raised to 1.2 MPa.

[0385] When the transfer members were observed through a fiberscope after 16 days of operation, the liquid contact portions of the reaction mixture excluding the valve portions maintained almost the same metal gloss as that before operation. However, when the valve was disassembled and inspected, white and brown products were adhered to the rear side of the disk of the valve and a residue portion formed in the rear side of the disk of the valve in large quantities. A brown product was partially adhered to a location where the flow of the valve sharply changed.

[0386] In the industrial process for producing a polycarbonate by melt polymerization, the retention of the reaction mixture is suppressed, the formation of a deteriorated product of the polymer is minimized, and a high-quality polycarbonate can be obtained as a base material for optical recording media by using (1) valves, (2) gear pumps, (3) flanges and (4) flow passages having specific structures and characteristic properties.

Claims

1. A process for producing a polycarbonate resin through the melt polycondensation of an aromatic diol compound and a carbonic acid diester compound, wherein
(1) valves, (2) pumps, (3) flanges and (4) flow

passages having the following structures and characteristic properties are used in the production process:

(1) valve: Y-shaped globe valve which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less and a pressure durability of 0.1 MPa or more, and whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound;

(2) gear pump: gear pump which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa, a pressure durability of 0.1 MPa or more and a delivery pressure of 1 MPa or more, whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound, and which has such a structure that the reaction mixture under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less can be discharged and that the reaction mixture for lubricating contact portions among a gear, shaft and other gear pump members does not return into the pump and is discharged to the outside of the pump;

(3) flange: flange which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less and a pressure durability of 0.1 MPa or more, whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound, and which has a heating medium jacket therein; and

(4) flow passage: flow passage which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less and a pressure durability of 0.1 MPa or more, whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound, which is restricted and/or expanded at a vertical angle of 20° or less, and whose surface is smooth without level differences.

55 2. A process for producing a polycarbonate resin through the melt polycondensation of an aromatic diol compound and a carbonic acid diester compound, wherein

(1) valves, (2) gear pumps and (3) flanges having the following structures and characteristic properties are used in the production process:

(1) valve: Y-shaped globe valve which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less and a pressure durability of 0.1 MPa or more, and whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound; 5

(2) gear pump: gear pump which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa, a pressure durability of 0.1 MPa or more and a delivery pressure of 1 MPa or more, whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound, and which has such a structure that the reaction mixture under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less can be discharged and that the reaction mixture for lubricating contact portions among a gear, shaft and other gear pump members does not return into the pump and is discharged to the outside of the pump; and 10

(3) flange: flange which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less and a pressure durability of 0.1 MPa or more, whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound, and which has a heating medium jacket therein. 15

3. A process for producing a polycarbonate resin through the melt polycondensation of an aromatic diol compound and a carbonic acid diester compound, wherein 20

at least two members out of (1) valves, (2) gear pumps and (3) flanges used in the production process have the following structures and characteristic properties:

(1) valve: Y-shaped globe valve which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less and a pressure durability of 0.1 MPa or more, and whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound 25

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formed by a reaction between the aromatic diol compound and the carbonic acid diester compound;

(2) gear pump: gear pump which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa, a pressure durability of 0.1 MPa or more and a delivery pressure of 1 MPa or more, whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound, and which has such a structure that the reaction mixture under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less can be discharged and that the reaction mixture for lubricating contact portions among a gear, shaft and other gear pump members does not return into the pump and is discharged to the outside of the pump; and 30

(3) flange: flange which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less and a pressure durability of 0.1 MPa or more, whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound, and which has a heating medium jacket therein. 35

4. The polycarbonate resin production process according to claim 3, wherein flow passages (4) having the following structure and characteristic properties are used:

(4) flow passage: flow passage which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less and a pressure durability of 0.1 MPa or more, whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound, which is restricted and/or expanded at a vertical angle of 20° or less, and whose surface is smooth without level differences. 45

5. A process for producing a polycarbonate resin through the melt polycondensation of an aromatic diol compound and a carbonic acid diester compound, wherein 50

at least one member out of (1) valves, (2) gear pumps and (3) flanges used in the production process has the following structure and characteristic properties:

(1) valve: Y-shaped globe valve which has a heat durability of 150°C or more, substantially 55

no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less and a pressure durability of 0.1 MPa or more, and whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound; 5

(2) gear pump; gear pump which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa, a pressure durability of 0.1 MPa or more and a delivery pressure of 1 MPa or more, whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound, and which has such a structure that the reaction mixture under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less can be discharged and that the reaction mixture for lubricating contact portions among a gear, shaft and other gear pump members does not return into the pump and is discharged to the outside of the pump; and 10

(3) flange; flange which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less and a pressure durability of 0.1 MPa or more, whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound, and which has such a structure that the reaction mixture under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less can be discharged and that the reaction mixture for lubricating contact portions among a gear, shaft and other gear pump members does not return into the pump and is discharged to the outside of the pump; 15

(3) flange; flange which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less and a pressure durability of 0.1 MPa or more, whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound, and which has such a structure that the reaction mixture under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less can be discharged and that the reaction mixture for lubricating contact portions among a gear, shaft and other gear pump members does not return into the pump and is discharged to the outside of the pump; 20

6. The polycarbonate resin production process according to claim 5, wherein flow passages (4) having the following structure and characteristic properties are used: 25

(4) flow passage; flow passage which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less and a pressure durability of 0.1 MPa or more, whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound, and which has a heating medium jacket therein. 30

7. A process for producing a polycarbonate resin through the melt polycondensation of an aromatic diol compound and a carbonic acid diester compound, wherein 35

at least one member out of (1) valves, (2) gear pumps, (3) flanges and (4) flow passages used in

the production process has the following structure and characteristic properties:

(1) valve; Y-shaped globe valve which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less and a pressure durability of 0.1 MPa or more, and whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound; 40

(2) gear pump; gear pump which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa, a pressure durability of 0.1 MPa or more and a delivery pressure of 1 MPa or more, whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound, and which has such a structure that the reaction mixture under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less can be discharged and that the reaction mixture for lubricating contact portions among a gear, shaft and other gear pump members does not return into the pump and is discharged to the outside of the pump; 45

(3) flange; flange which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less and a pressure durability of 0.1 MPa or more, whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound, and which has a heating medium jacket therein; and 50

(4) flow passage; flow passage which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of $40,000 \times 10^{-6}$ MPa or less and a pressure durability of 0.1 MPa or more, whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound, and which is restricted and/or expanded at an vertical angle of 20° or less, and whose surface is smooth without level differences. 55

8. A process for producing a polycarbonate resin through the melt polycondensation of an aromatic diol compound and a carbonic acid diester compound, wherein

(1) valves having the following structure and

characteristic properties are used in the production process:

(1) valve; Y-shaped globe valve which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of 40,000 x 10⁻⁶ MPa or less and a pressure durability of 0.1 MPa or more, and whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound.

9. The polycarbonate production process according to claim 8, wherein the stem of the Y-shaped globe valve has substantially the same thickness as that of a portion into which the stem is inserted of a valve body.

10. The polycarbonate production process according to claim 8 or 9, wherein an end portion of the stem of the Y-shaped globe valve and the inner surface of an upper tubular portion of the valve form a smooth curved surface when the valve is opened.

11. The polycarbonate production process according to any one of claims 8 to 10, wherein an O ring is attached to the stem of the Y-shaped globe valve to seal a gap between the stem and the portion into which the stem is inserted of the valve body.

12. The polycarbonate production process according to any one of claims 8 to 11, wherein the liquid contact portion of the Y-shaped globe valve has a surface roughness of 10 pm or less.

13. A process for producing a polycarbonate resin through the melt polycondensation of an aromatic diol compound and a carbonic acid diester compound, wherein

(2) gear pumps having the following structure and characteristic properties are used in the production process:

(2) gear pump; gear pump which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of 40,000 x 10⁻⁶ MPa, a pressure durability of 0.1 MPa or more and a delivery pressure of 1 MPa or more, whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound, and which has such a structure that the reaction mixture under a vacuum pressure of 40,000 x 10⁻⁶ MPa or less can be discharged and that the reaction mixture

for lubricating contact portions among a gear, shaft and other gear pump members does not return into the pump and is discharged to the outside of the pump.

5 14. The polycarbonate resin production process according to claim 13, wherein the gear pump comprises a scraping blade for preventing the reaction mixture from residing in a shaft end portion of the gear.

15. The polycarbonate resin production process according to claim 13 or 14, wherein a bushing for supporting the shaft of the gear is fixed to a cover plate and there is substantially no space between the cover plate and the bushing in the gear pump.

16. The polycarbonate resin production process according to any one of claims 13 to 15, wherein a flow passage at the reaction mixture inlet port of the gear pump has a restriction angle of 45° or less and has a smooth surface without level differences.

20 17. The polycarbonate resin production process according to any one of claims 13 to 16, wherein the liquid contact portion of the gear pump has a surface roughness of 10 µm or less.

25 18. The polycarbonate resin production process according to any one of claims 13 to 17, wherein a gland packing is used as a shaft seal material for the driving shaft of the gear pump.

30 19. The polycarbonate resin production process according to any one of claims 13 to 17, wherein a labyrinth seal is used as a shaft seal material for the driving shaft of the gear pump.

35 20. The polycarbonate resin production process according to any one of claims 13 to 19, wherein a hollow metal O ring is used to seal the fixed portion of the gear pump.

40 21. A process for producing a polycarbonate resin through the melt polycondensation of an aromatic diol compound and a carbonic acid diester compound, wherein

45 (3) flanges having the following structure and characteristic properties are used in the production process:

50 (3) flange; flange which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of 40,000 x 10⁻⁶ MPa or less and a pressure durability of 0.1 MPa or more, whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction

between the aromatic diol compound and the carbonic acid diester compound, and which has a heating medium jacket therein.

22. The polycarbonate resin production process according to claim 21, wherein the flange has a groove-shaped gasket seal. 5

23. The polycarbonate resin production process according to claim 21 or 22, wherein a gasket for connecting the flange has a metal O ring. 10

24. The polycarbonate resin production process according to any one of claims 21 to 23, wherein a spacer is sandwiched between the flanges. 15

25. The polycarbonate resin production process according to any one of claims 21 to 24, wherein the liquid contact portion of the flange has a surface roughness of 10 µm or less. 20

26. A process for producing a polycarbonate resin through the melt polycondensation of an aromatic diol compound and a carbonic acid diester compound, wherein 25
(4) flow passages having the following structure and characteristic properties are used in the production process:
(4) flow passage; flow passage which has a heat durability of 150°C or more, substantially no leakage under a vacuum pressure of 40,000 x 10⁻⁶ MPa or less and a pressure durability of 0.1 MPa or more, whose liquid contact portion is made from a material having corrosion resistance against a monohydroxy compound formed by a reaction between the aromatic diol compound and the carbonic acid diester compound, which is restricted and/or expanded at a vertical angle of 20° or less, and whose surface is smooth without level differences. 30
35
40

27. The polycarbonate resin production process according to claim 26, wherein the restricted and/or expanded portion of the flow passage is a reducer. 45

28. The polycarbonate resin production process according to claim 26, wherein the flow passage is a pipe. 50

29. The polycarbonate resin production process according to claim 28, wherein the curvature of a curved portion of the pipe is 5 times or more the inner diameter of the pipe. 55

30. The polycarbonate resin production process according to claim 28, wherein the pipe is connected by a socket.

31. The polycarbonate resin production process according to claim 26, wherein the liquid contact portion of the flow passage has a surface roughness of 10 µm or less.

FIG. I

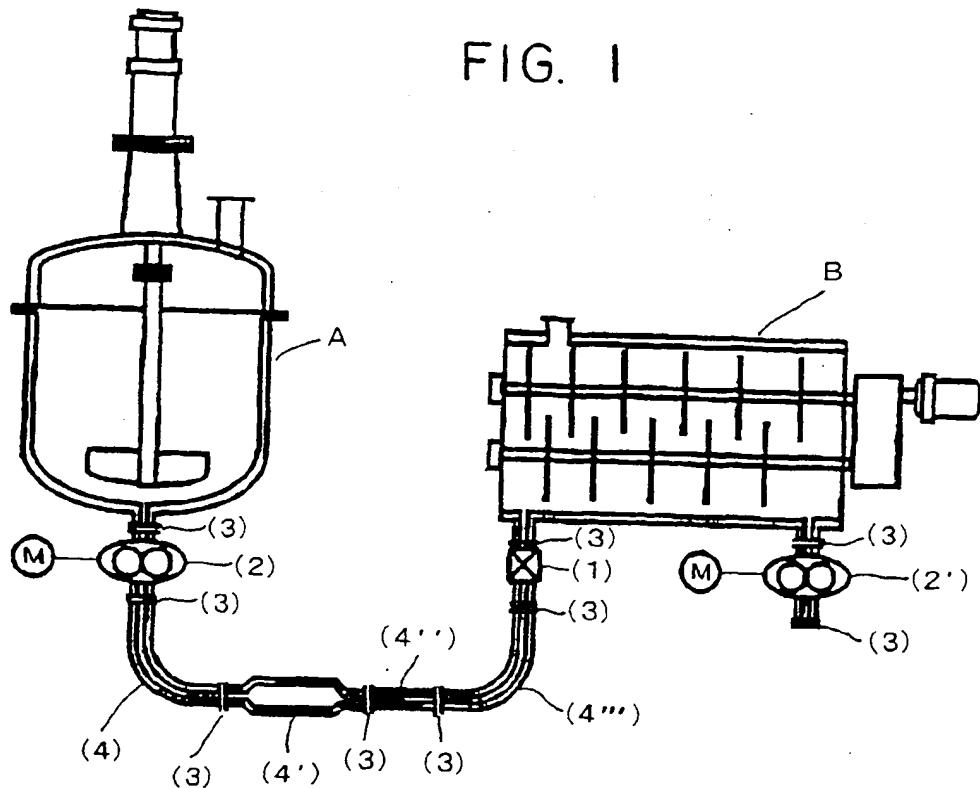


FIG. 2

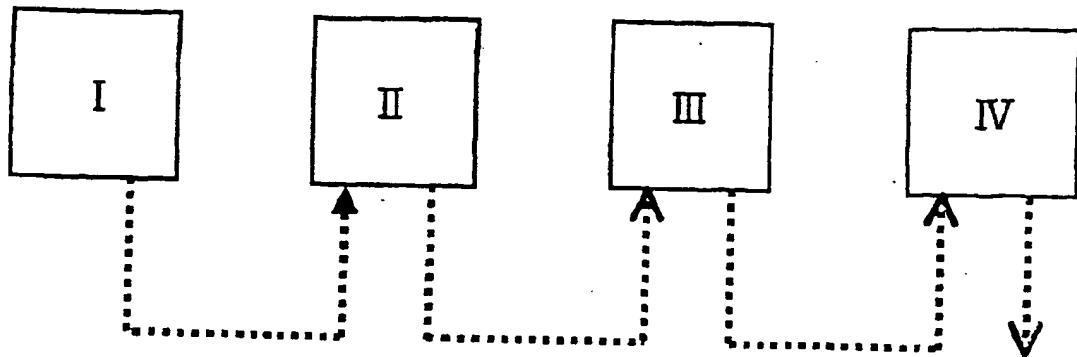


FIG. 3

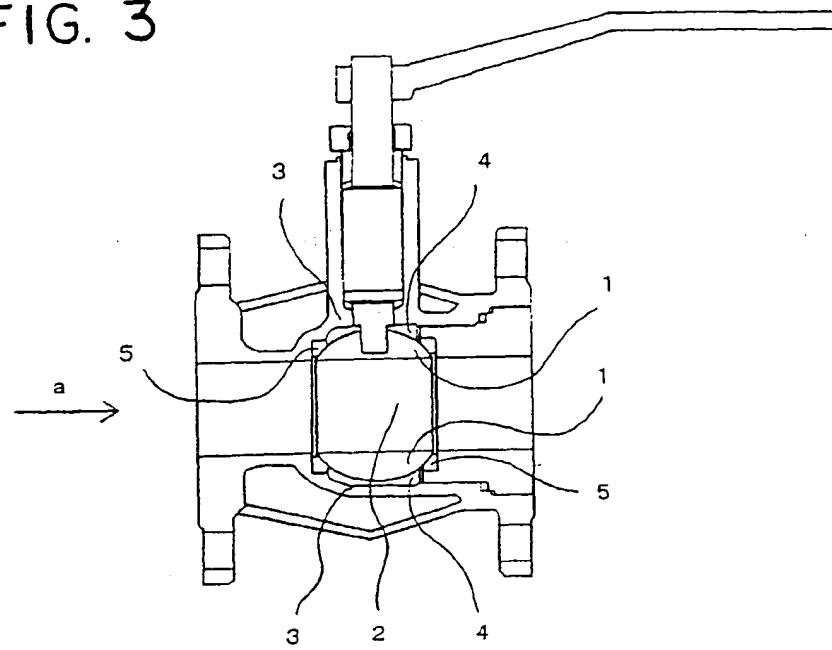


FIG. 4

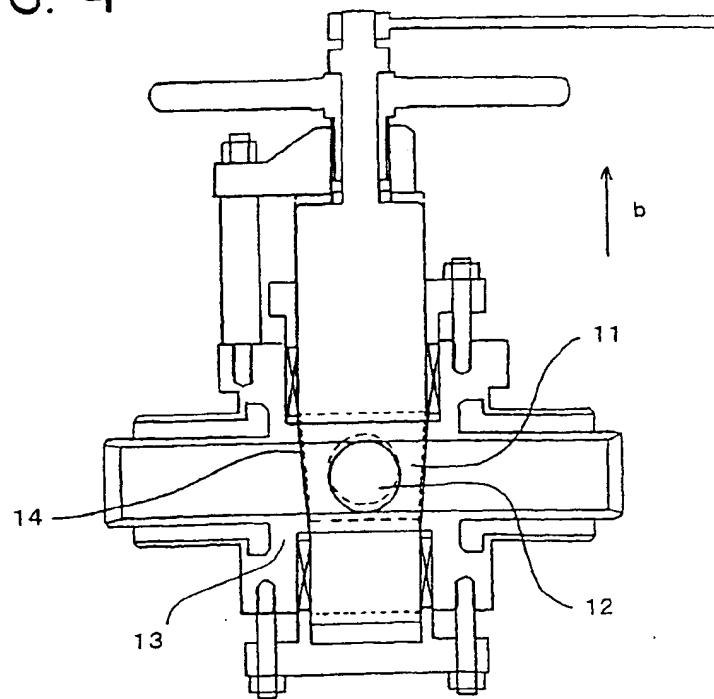


FIG. 5

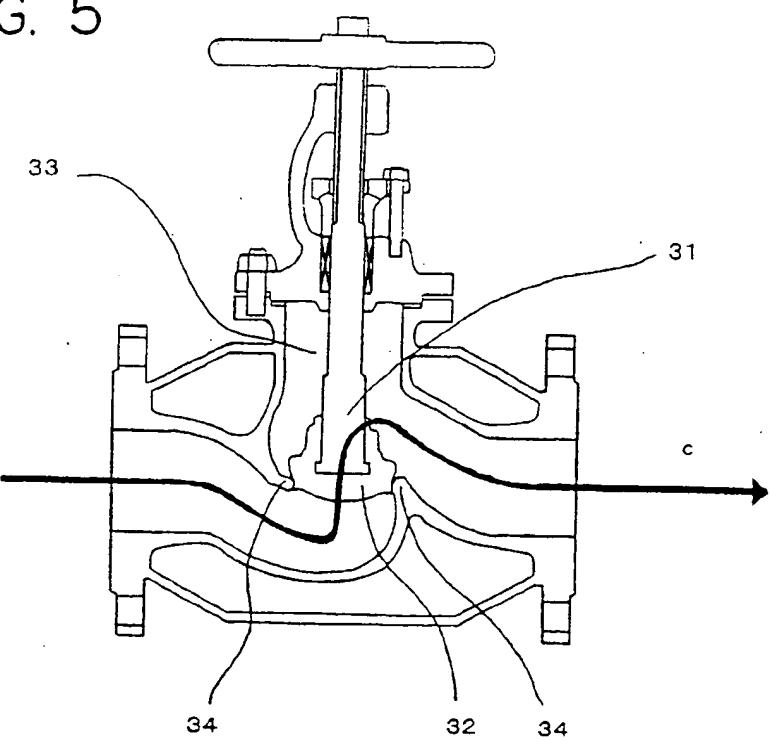


FIG. 6

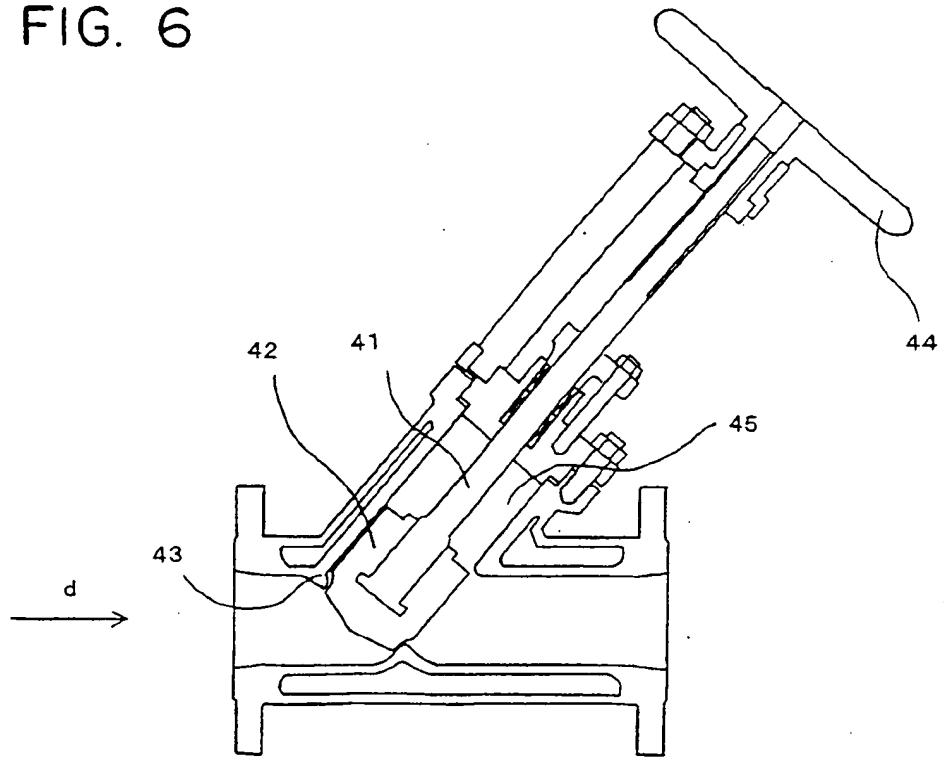


FIG. 7

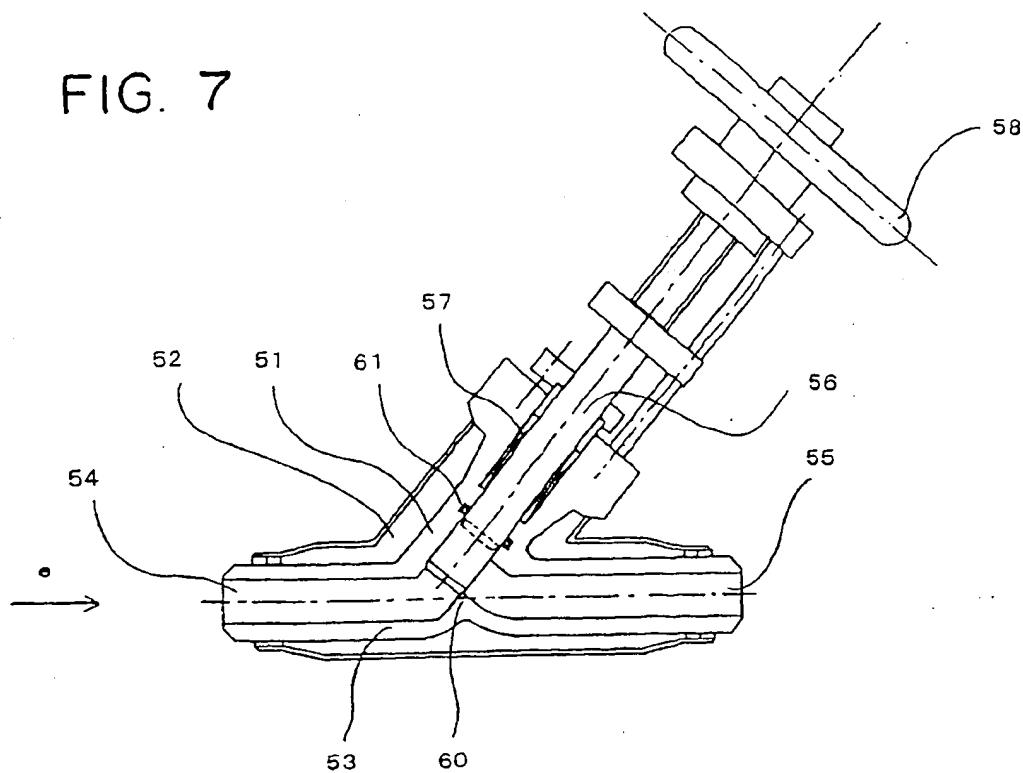


FIG. 8

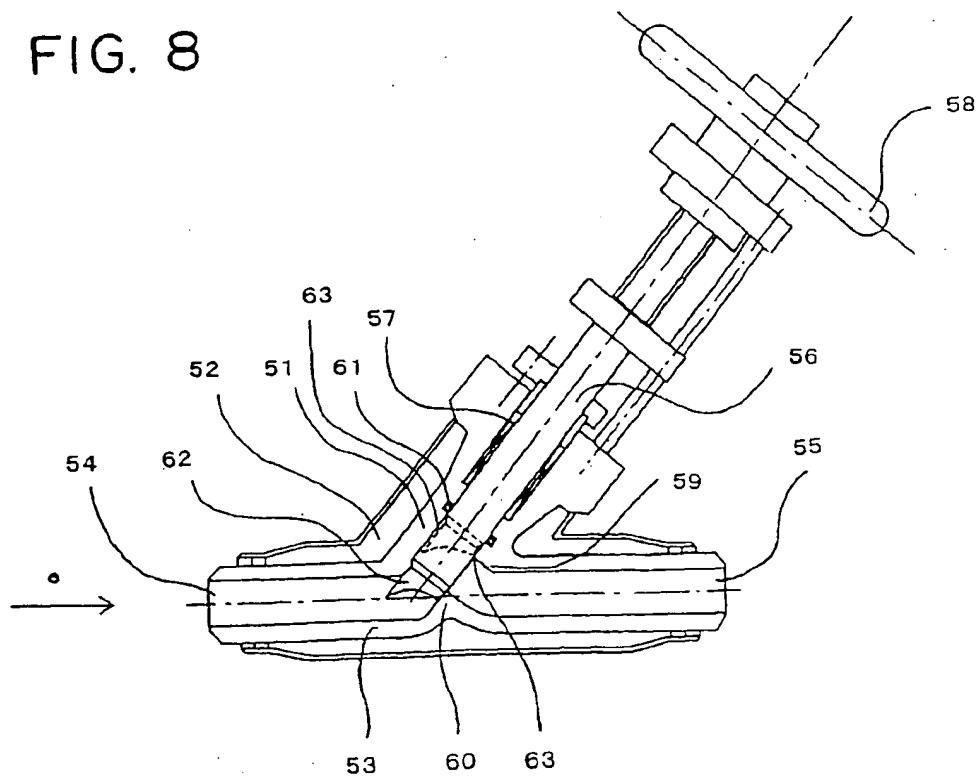


FIG. 9

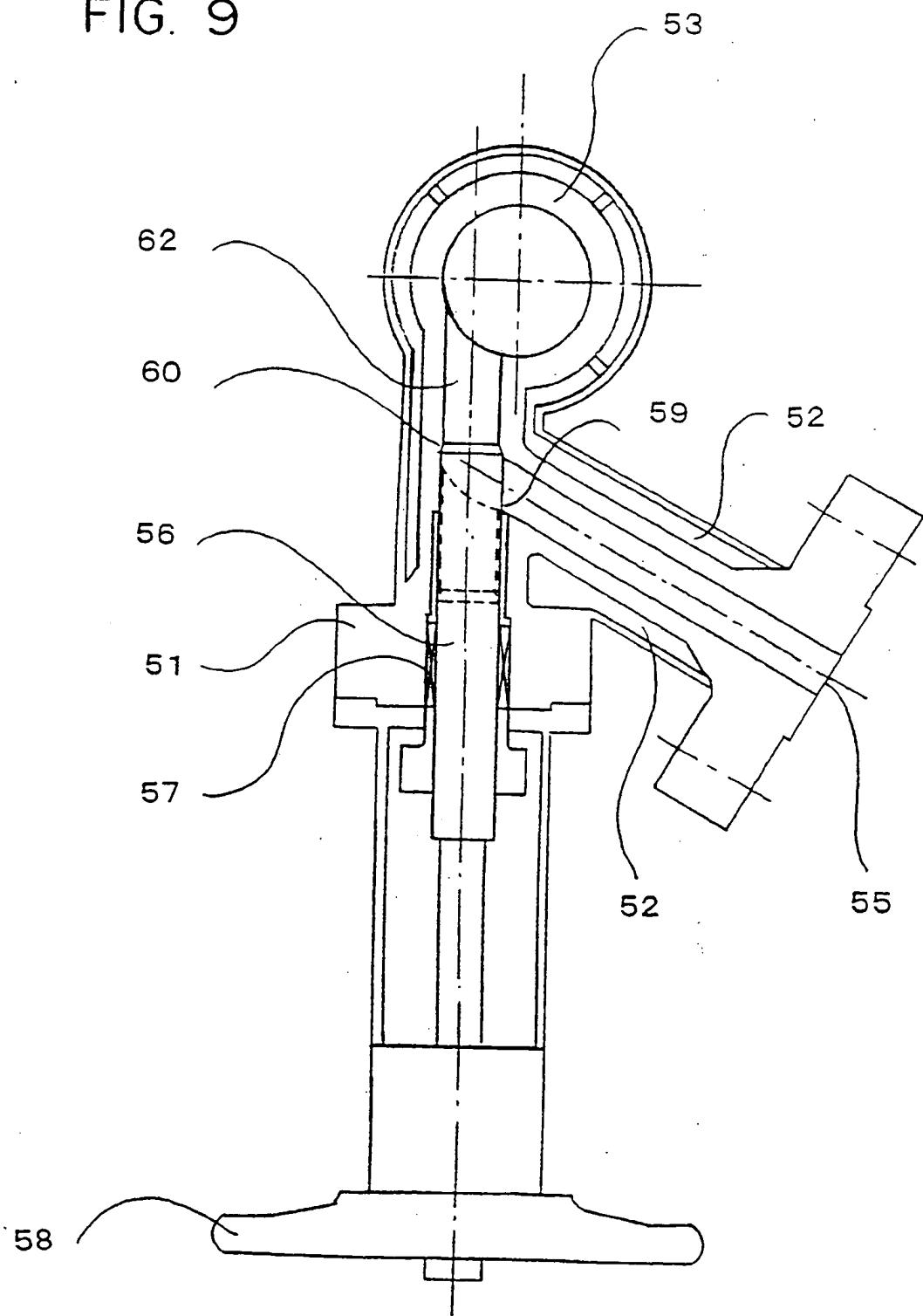


FIG. 10

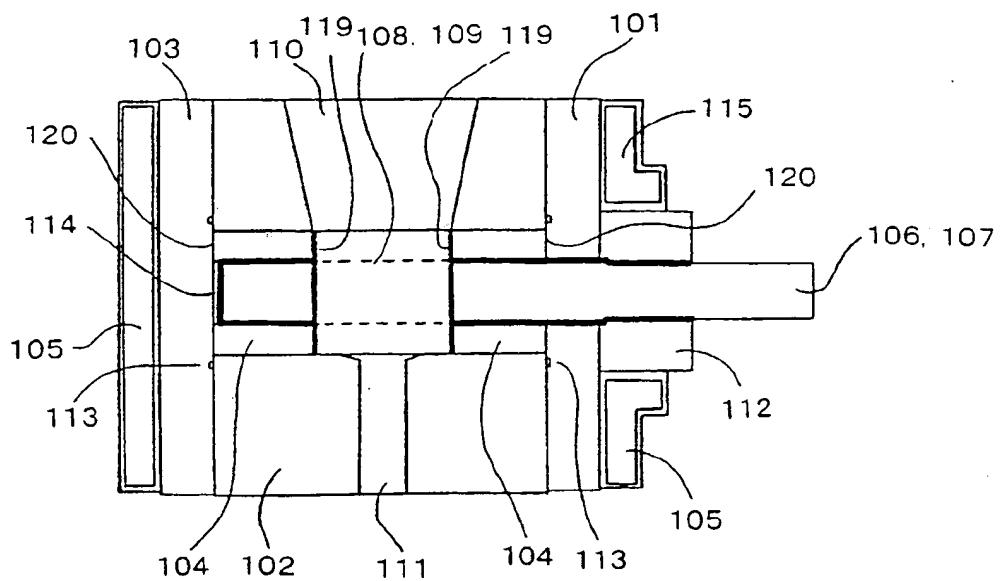


FIG. 11

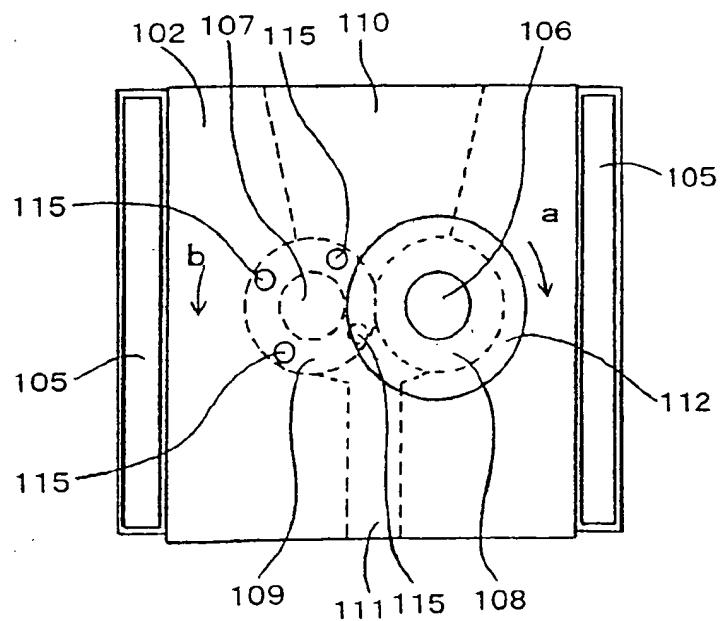


FIG. 12

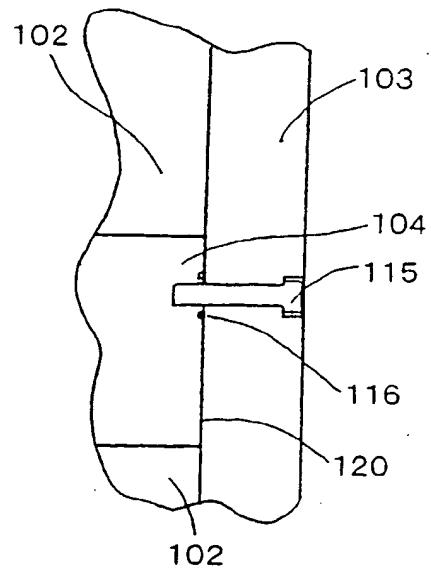


FIG. 13

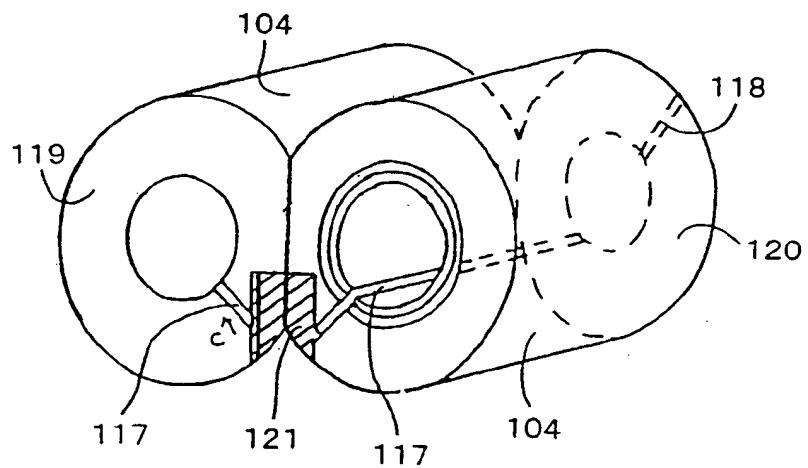


FIG. 14

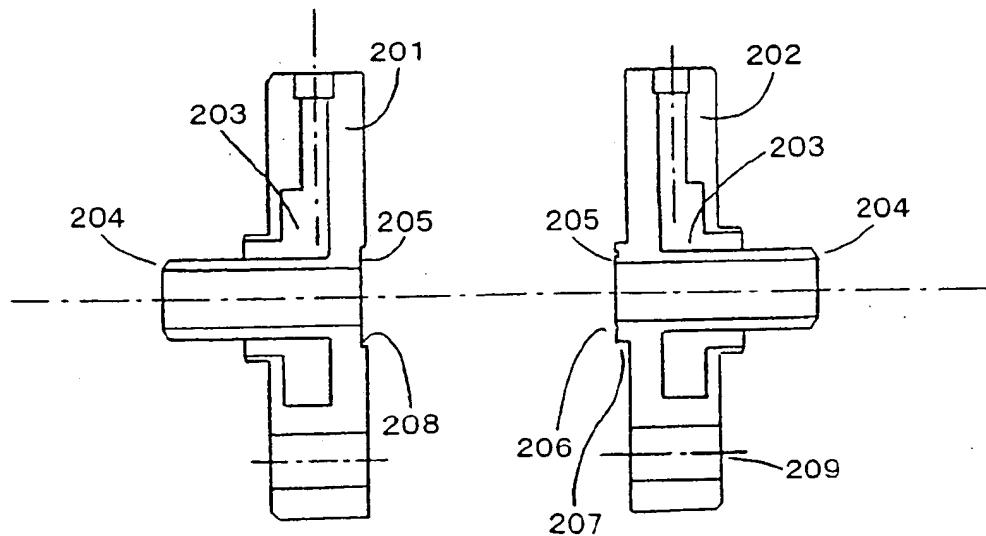


FIG. 15

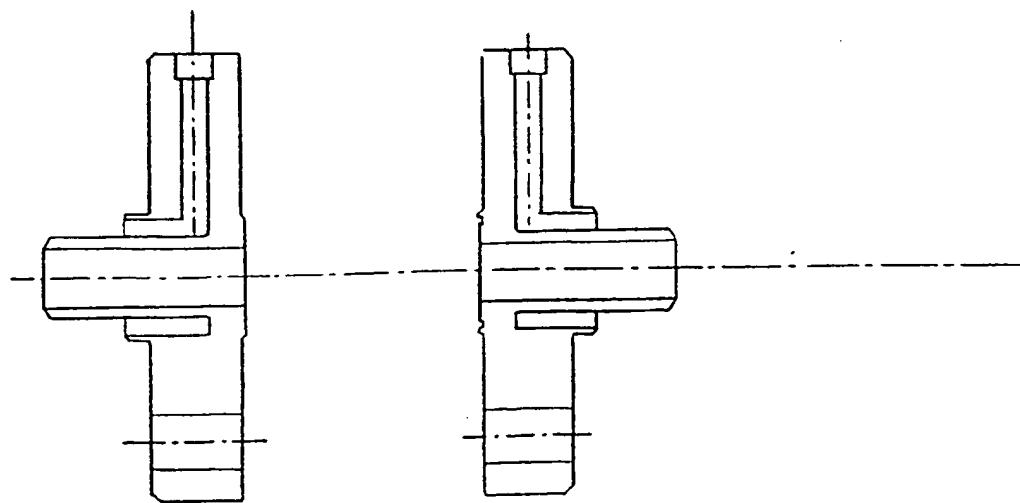


FIG. 16

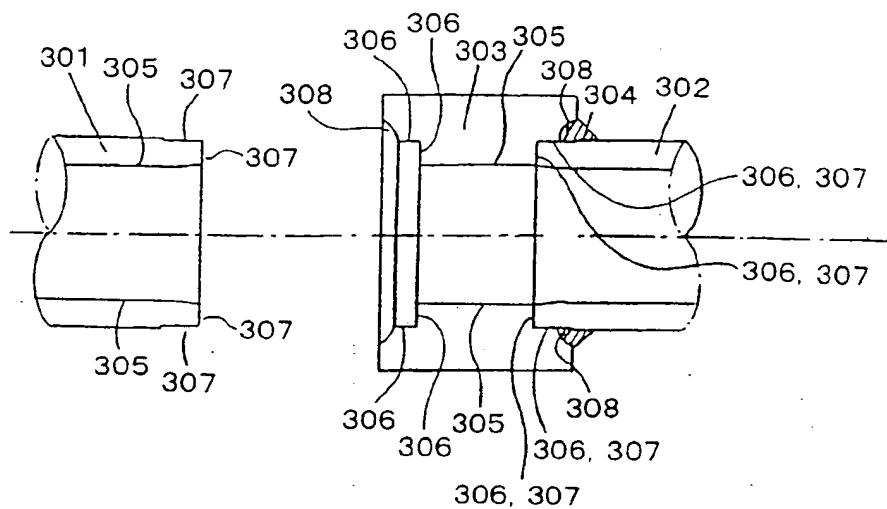


FIG. 21

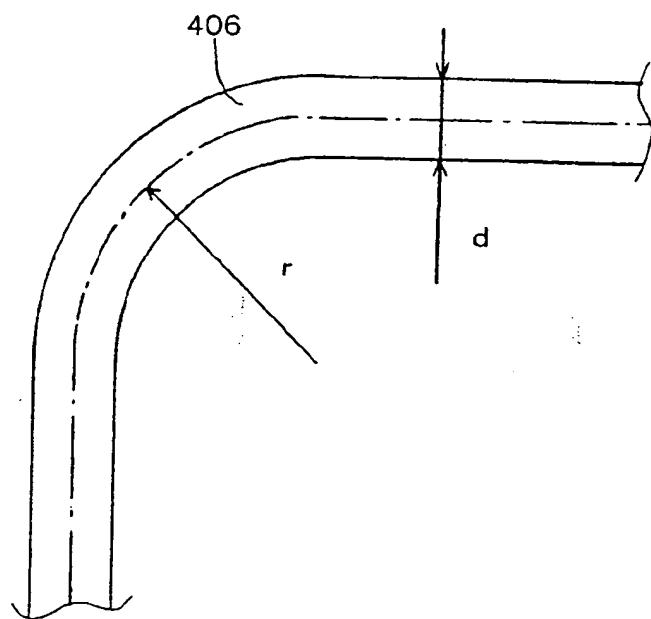


FIG. 17

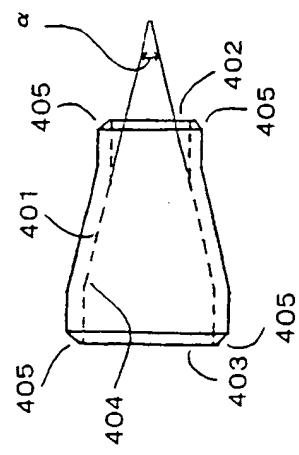


FIG. 19

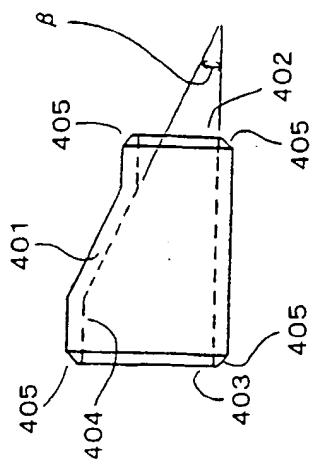


FIG. 18

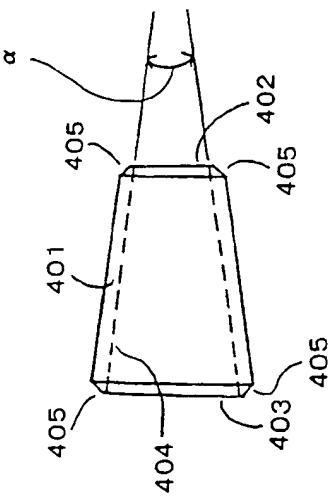
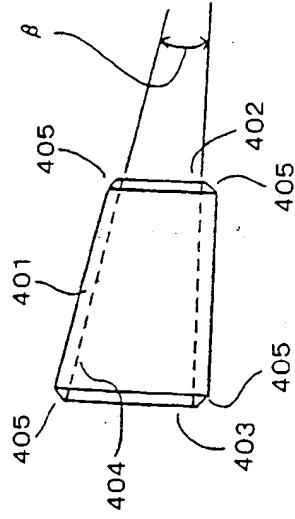


FIG. 20





(19)

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(11)

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(12)

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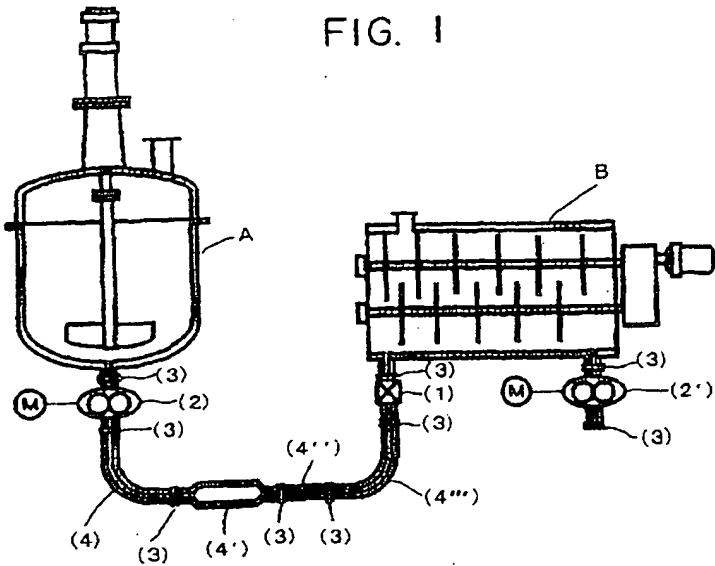
(54) Process for producing a polycarbonate resin

(57) A process for producing a polycarbonate resin through the melt polycondensation of an aromatic diol compound and a carbonic acid diester compound, wherein

(1) valves, (2) gear pumps, (3) flanges and (4) flow passageways having the specified structures and characteristic properties are used in the production process.

According to the present invention, a high-quality polycarbonate can be obtained by an industrial process.

FIG. 1



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European Patent
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EUROPEAN SEARCH REPORT

Application Number
EP 99 30 4360

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
A	EP 0 601 602 A (DAICEL CHEM) 15 June 1994 (1994-06-15) * page 7, line 10-58; example 1 *	1	C08G64/20 C08G64/30
TECHNICAL FIELDS SEARCHED (Int.Cl.6)			
C08G			
The present search report has been drawn up for all claims			
Place of search	Date of completion of the search	Examiner	
MUNICH	22 May 2001	Von Kuzenko, M	
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ON EUROPEAN PATENT APPLICATION NO.

EP 99 30 4360

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22-05-2001

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